CHEM 3P20
Library Seminar
Fall 2024

Ian Gordon

Ian Gordon, Teaching & Learning Librarian

Brock University Library

CHEM 3P20 Total Synthesis Term Project

For the term project, students are to come up with a synthetic plan for a molecule of interest and/or importance. You will propose a novel total synthesis (cannot be copied from literature, has to be an original synthetic plan) of this molecule. The synthetic targets should be ideally from the field of medicinal chemistry or natural products. Once the desired molecule is selected, come up with a rationale for why the molecule is synthesized and reason for your interest. The point of this project is to get creative with your knowledge of chemistry and apply what you have learned so far in your academics.

Project Requirements:

1) The molecule must have five stereochemical centers, ideally chiral, but not necessary
    a. Can be a racemic mixture
    b. Must be in the field of medicinal chemistry or natural product
2) There cannot be more than 8 linear steps in synthesis (can have convergent steps)
3) Cannot be a cyclic peptide
4) The synthetic procedure cannot be taken directly from literature. I.e. you cannot present a previously known synthesis as your own.
    a. You can borrow certain segments
5) Email your synthetic targets to your TA as soon as possible for approval. Try to have 3 potential targets.

Presentation Requirements (60%)

1) You and your partner will present a 25-30 min presentation of your chosen total synthesis during lecture. Presentations will start _____. Following the presentations, there will be a short round for questions.
    a. Presentation will include a brief history of the molecule, importance/background, and use in respective field.
    b. Followed by your novel synthesis and rationale for each step.
2) The presentation slides must be sent to Dr. Dudding.

Paper requirements (40%)

By the last day of the semester, you should turn in a 4-5-page paper discussing your synthesis. This essentially covers what was discussed in your presentation. Details regarding the paper will be covered in class.

Library Seminar Agenda

- CHEM Library Research Guide
- Databases – lots of them!
- Citation management
- Where, how and when to get help

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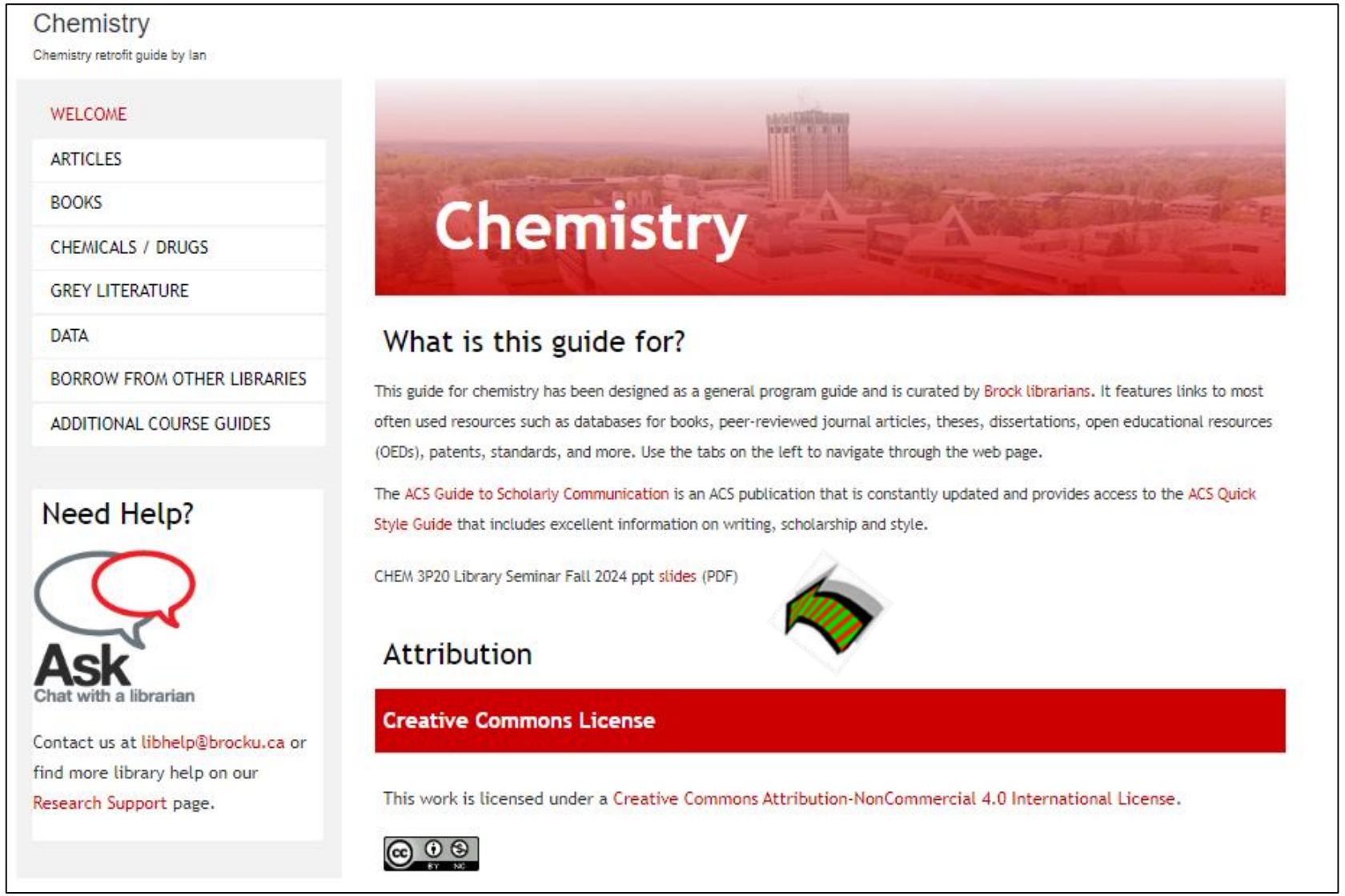
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The ACS Guide to Scholarly Communication is an ACS publication that is constantly updated and provides access to the ACS Quick Style Guide that includes excellent information on writing, scholarship and style.
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Articles

Journal articles are scholarly works that go through a quality control process called peer-review before they are published.

Several databases provide access to regional, national and international news articles.

A select list of databases that include scholarly articles are listed below.

The ACS Guide to Scholarly Communication is an ACS publication that is constantly updated and provides access to the ACS Quick Style Guide that includes excellent information on writing, scholar

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- Google Scholar
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- Web of Science Core Collection
  - A multidisciplinary index of citations to articles in finding journal articles published on a topic or to
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more info...

- SciFinder-n
  - Core chemistry, medicine, health, environmental and scientific journals, patents from international agencies, single and multi-step reactions, predicted and experimental property values, reports and dissertations from 1800s-present.
  - SciFinder-n is a completely different and updated interface to the classic CAS SciFinder database, with improved search algorithm and added features
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  - For access on and off campus - register for account, create username & password, confirm registration by e-mail
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more info...

- Reaxys
  - Find chemical structures, reactions, and properties.
  - Useful for researching synthetic methods of chemical compounds, characterizing synthesized compounds, or searching for critically evaluated data on physical, chemical, spectral, bioactivity, or toxicological properties.
  - Includes Beilstein (to 1959, Supplement IV); Gmelin (to 1975); and selected organic chemistry patents (1869-1980) with English-language chemical patents (US, WO, EP, 1976+) from selected primary IPC classes.
  - For full usability set up an individual account for access on and off campus. Java and JRE plugins may be required.
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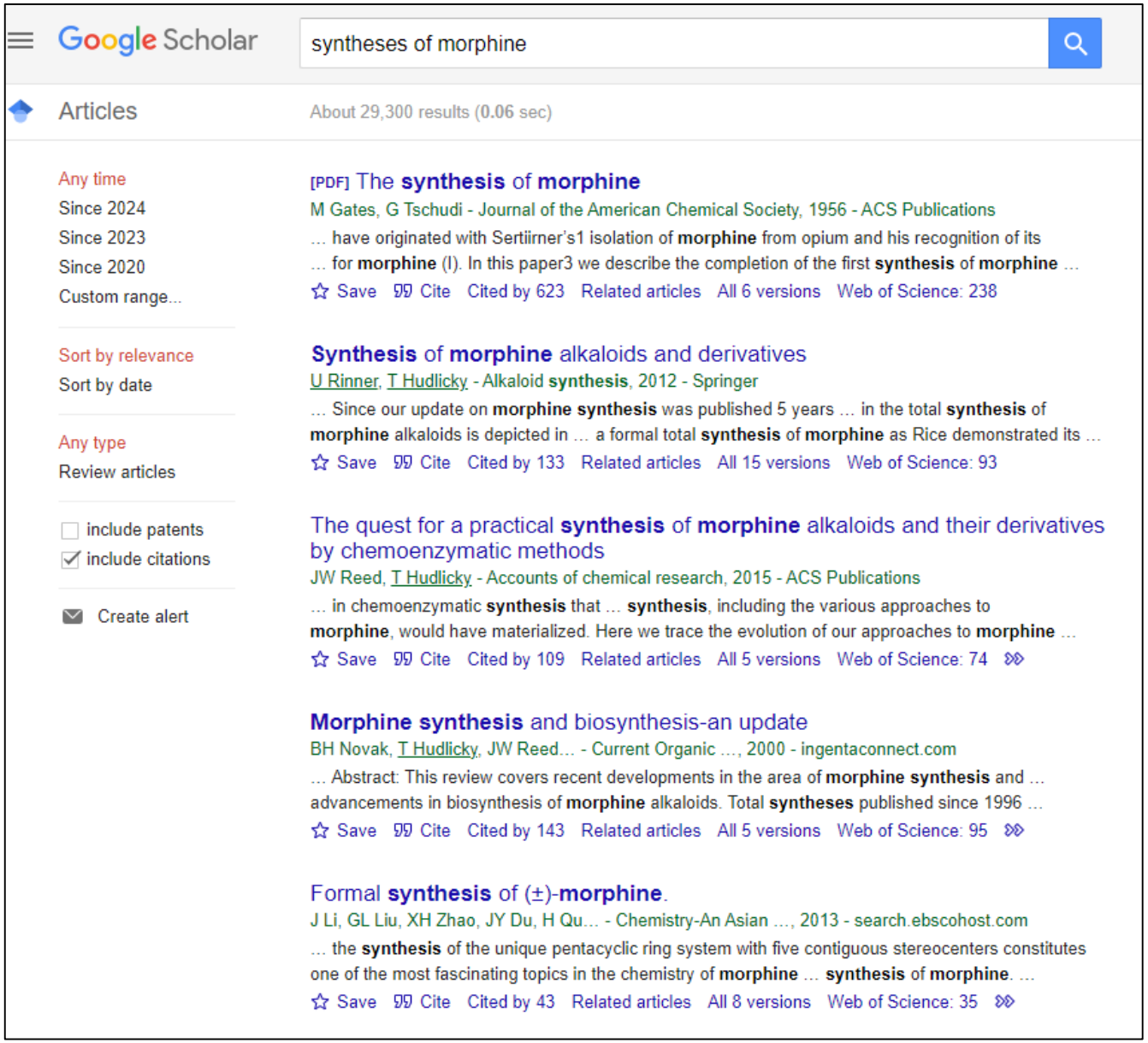
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[PDF] The synthesis of morphine
M Gates, G Tschudi - Journal of the American Chemical Society, 1956 - ACS Publications
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... for morphine (I). In this paper3 we describe the completion of the first synthesis of morphine ...
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Synthesis of morphine alkaloids and derivatives
U Rinner, T Hudlicky - Alkaloid synthesis, 2012 - Springer
... Since our update on morphine synthesis was published 5 years ... in the total synthesis of
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The quest for a practical synthesis of morphine alkaloids and their derivatives by chemoenzymatic methods
JW Reed, T Hudlicky - Accounts of chemical research, 2015 - ACS Publications
... in chemoenzymatic synthesis that ... synthesis, including the various approaches to
morphine, would have materialized. Here we trace the evolution of our approaches to morphine ...
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Morphine synthesis and biosynthesis-an update
BH Novak, T Hudlicky, JW Reed... - Current Organic ..., 2000 - ingentaconnect.com
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Formal synthesis of (±)-morphine.
J Li, GL Liu, XH Zhao, JY Du, H Qu... - Chemistry-An Asian ..., 2013 - search.ebscohost.com
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one of the most fascinating topics in the chemistry of morphine ... synthesis of morphine. ...
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Research Topic:

I want to find scholarly articles that focus on synthetic approaches to morphine, detailing specific experimental methods, catalysts, or novel pathways used in its synthesis.

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Detailed summary

A plethora of recent studies have centered on various synthetic approaches to morphine, utilizing novel pathways and catalysts to achieve efficiency and stereochemical control [1, 2, 3, 4].

Key Insights on Morphine Synthesis:

- **Novel Pathways and Strategies:**
  - Recent syntheses have employed innovative techniques such as oxidative phenolic coupling and
  - Biomimetic and chemoenzymatic methods have been explored, providing alternative strategies th
- **Catalysts and Reagents:**
  - Asymmetric hydrogenations with organometallic catalysts have been pivotal, with advances in ca
  - Enzymatic strategies and chemoenzymatic approaches, involving genetically engineered enzyme
- **Efficiency and Stereoselectivity:**
  - Efficient total syntheses have been achieved with reduced steps and improved overall yields, leve
  - Innovations in stereoselective transformations, such as sequential Claisen rearrangements, have

These findings highlight ongoing advancements in the field, focusing on optimizing reaction conditions a

Categories of papers

Timeline and citation network

Discovery progress: ~52.2% complete (~20-27 papers found)

References

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Topic Match	Cit./Year	Year	Paper
99.5%	3.9	2014	**[1] Gram-scale enantioselective formal synthesis of morphine through an ortho-para oxidative phenolic coupling strategy.** M. Tissot, ..., and M. Gaunt Angewandte Chemie 2014 - 38 citations - Show abstract - Cite
99.4%	3.0	2010	**[2] Total synthesis of (-)-morphine.** H. Koizumi, ..., and T. Fukuyama Chemistry, an Asian journal 2010 - 42 citations - Show abstract - Cite - PDF
99.3%	6.0	2002	**[3] Synthesis of (-)-morphine.** D. Taber, ..., and A. Rheingold Journal of the American Chemical Society 2002 - 132 citations - Show abstract - Cite
99.2%	2.8	2013	**[4] Synthesis of (-)-morphine: application of sequential Claisen/Claisen rearrangement of an allylic vicinal diol.** M. Ichiki, ..., and N. Chida Chemistry 2013 - 33 citations - Show abstract - Cite
99.1%	3.6	2017	**[5] Total Synthesis of (-)-Morphine.** Hirotatsu Umihara, ..., and T. Fukuyama Chemistry 2017 - 26 citations - Show abstract - Cite - PDF
99.1%	0.5	2006	**[6] Syntheses of Morphine and Codeine (1992 - 2002): Templates for Exploration of Synthetic Tools** L. Mascavage, ..., and D. R. Dalton Current Organic Synthesis 2006 - 9 citations - Show abstract - Cite - PDF
99.0%	0.3	2008	**[7] A Practical Asymmetric Synthesisof the ACNO Fragment of Morphine Alkaloids** L. Hsin, ..., and Huei-Ling Liou Synlett 2008 - 5 citations - Show abstract - Cite
98.9%	0.4	2006	**[8] Chemoenzymatic Synthesis of Complex Natural and Unnatural Products: Morphine, Pancratistatin and Their Analogues** T. Hudlický ChemInform 2006 - 7 citations - Show abstract - Cite
98.9%	7.0	2015	**[9] The quest for a practical synthesis of morphine alkaloids and their derivatives by chemoenzymatic methods.** J. W. Reed and T. Hudlický Accounts of chemical research 2015 - 67 citations - Show abstract - Cite

https://chatgpt.com/

top syntheses for morphine?

Morphine, an opiate used primarily for pain relief, is synthesized through various methods, but it's important to remember that handling and synthesizing controlled substances like morphine should be done only by qualified professionals in regulated environments due to legal and safety concerns.

Here are a few well-known synthetic approaches to morphine:

1. **Thebain-Based Synthesis**: The most common synthetic route starts from thebaine, a compound found in opium. The synthesis involves a series of chemical transformations, including:
   - **Hydrolysis** of thebaine to obtain oripavine.
   - **Reduction and rearrangement** to produce morphine.
2. **Total Synthesis from Simple Precursors**: This approach aims to construct morphine starting from simpler chemical entities. One notable method involves:
   - The use of intermediates like 3,6-dioxopiperidine and complex reactions to build the morphine structure from scratch.
3. **Semi-Synthesis from Codeine**: Codeine, another opiate present in opium, can be chemically modified to produce morphine:
   - **O-demethylation** of codeine can yield morphine through a series of reactions.
4. **Total Synthesis from Simple Chemicals**: This method involves building the morphine molecule entirely from simpler starting materials. A classic example is the synthesis developed by Robert Woodward and his team, which was one of the earliest total syntheses of morphine.
5. **Total Synthesis from Benzylisoquinoline**: This involves using benzylisoquinoline as a starting material and applying a series of chemical transformations to produce morphine.

These methods are typically complex and require a high level of expertise in organic chemistry. If you're interested in the detailed procedures, consulting scientific literature and chemical synthesis textbooks would be the best approach. Always ensure to follow legal regulations and safety guidelines when dealing with substances like m phine.

https://en.wikipedia.org/wiki/Total_synthesis_of_morphine_and_related_alkaloids

Total synthesis of morphine and related alkaloids

1 language

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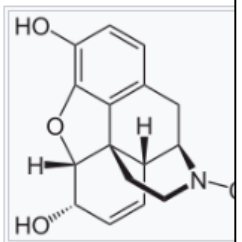
From Wikipedia, the free encyclopedia

Synthesis of morphine-like alkaloids in chemistry describes the total synthesis of the natural morphinan class of alkaloids that includes codeine, morphine, oripavine, and thebaine and the closely related semisynthetic analogs methorphan, buprenorphine, hydromorphone, hydrocodone, isocodeine, naltrexone, nalbuphine, oxymorphone, oxycodone, and naloxone.[1][2]



Morphine

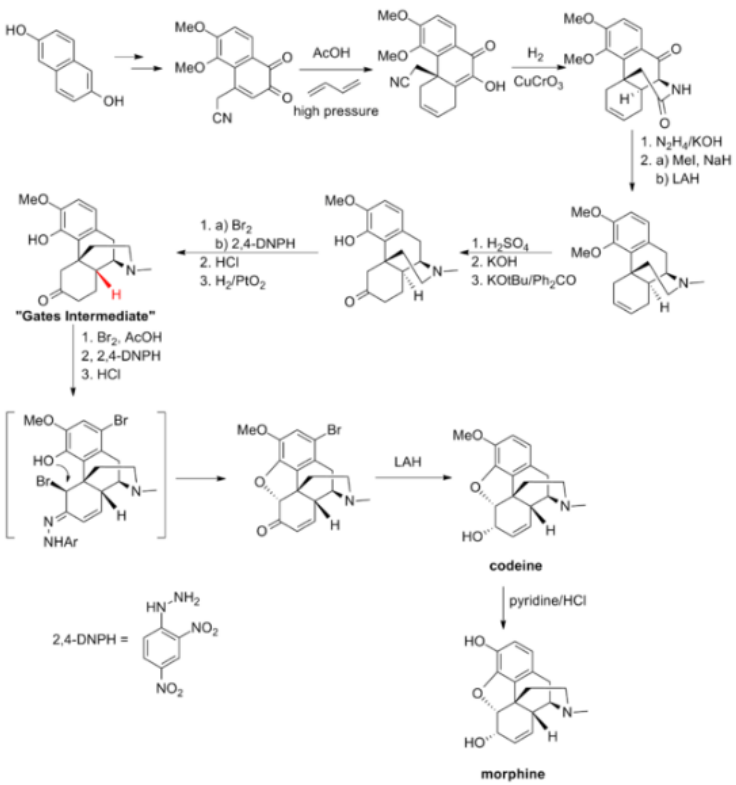
The structure of morphine is not particularly complex, however the electrostatic polarization of adjacent bonded atoms does not alternate uniformly throughout the structure. This "dissonant connectivity" makes bond formation more difficult and therefore significantly complicates any synthetic strategy that is applied to this family of molecules.[2]

The first morphine total synthesis, devised by Marshall D. Gates, Jr. in 1952 remains a widely used example of total synthesis.[3] synthesis took a total of 31 steps and proceeded in 0.06% overall yield. The hydrocodone synthesis of Kenner C. Rice is one of t most efficient and proceeds in 30% overall yield in 14 steps.[4] At 9 steps, the Barriault route is the shortest to date, but contains a number of low-yielding steps and is racemic.[5]

Several other syntheses were reported, notably by the research groups of Evans,[6] Fuchs,[7] Parker,[8] Overman,[9] Mulzer-Trauner,[10] White,[11] Taber,[12] Trost,[13] Fukuyama,[14] Guillou,[15] Stork,[16] Magnus,[17] Smith,[18] and Barriault.[5]

Gates synthesis [edit]

Gates' total synthesis of morphine[3] provided a proof of the structure of morphine proposed by Robinson in 1925.[19] morphine features one of the first examples of the Diels-Alder reaction in the context of total synthesis.[3]



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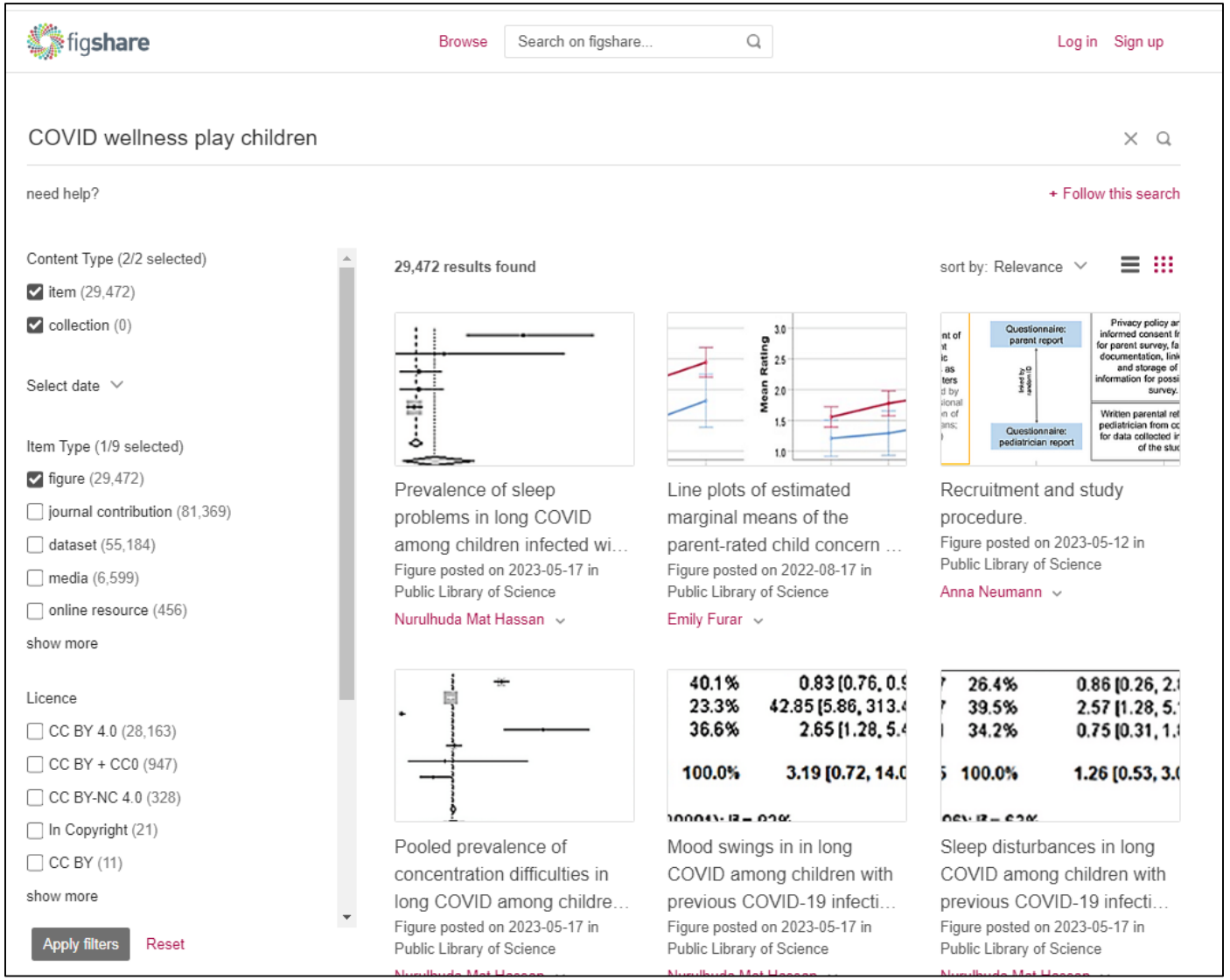
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Preparation of Highly-Substituted Pyridines via Diels-Alder Reactions of Vinylallenes and Tosyl Cyanide

Nathan H. Faialaga, Christian Gomez, Samuel G. Bartko, Philipp Natho, and Rick L. Danheiser

Org. Synth. **2023**, *100*, 4
DOI: 10.15227/orgsyn.100.0004

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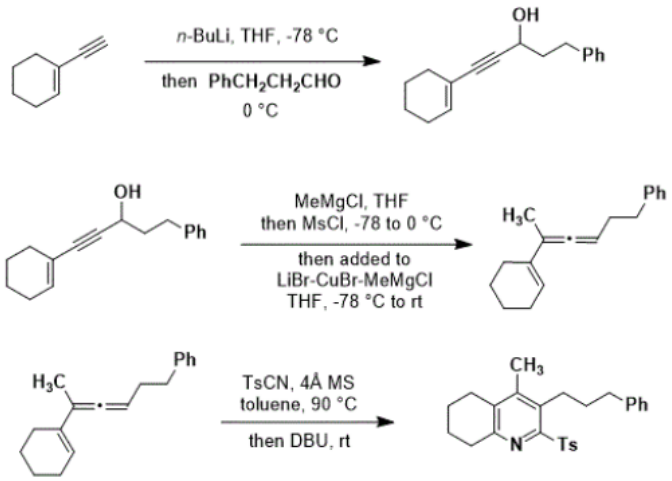

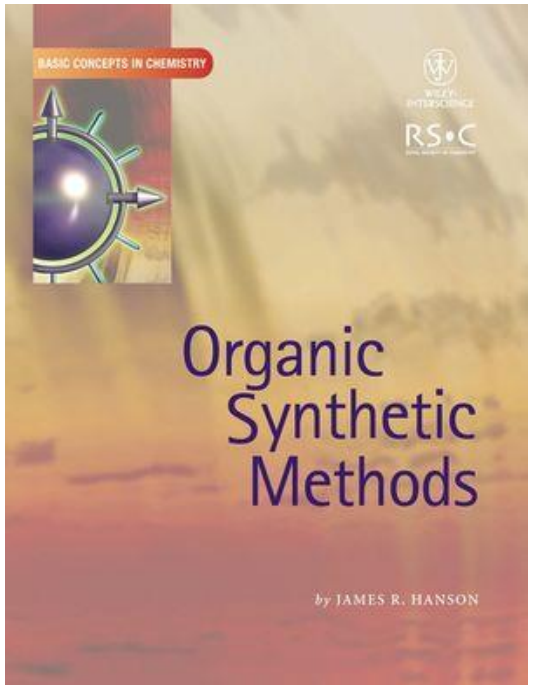
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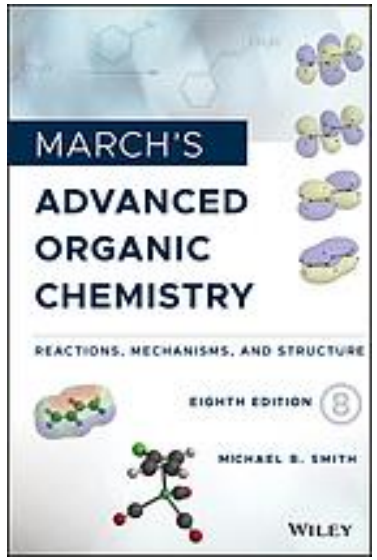
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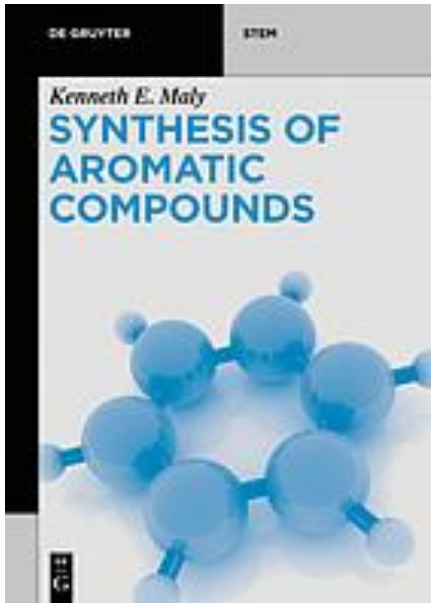
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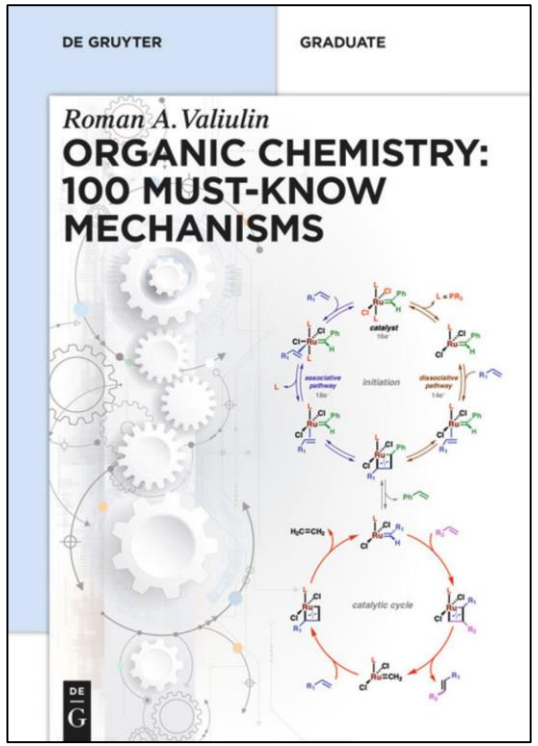
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Definitions are scholarly dictionaries, encyclopedias and handbooks that help define terms and provide additional context.

Dictionary of Chemistry (2020) Biochemistry and Molecular Biology (2006) Biomedicine (2010)

Taber's Cyclopedic Medical Dictionary (2021)

Compendium of Chemical Terminology (current, IUPAC)

CRC Handbook of Chemistry & Physics (2014/2015)

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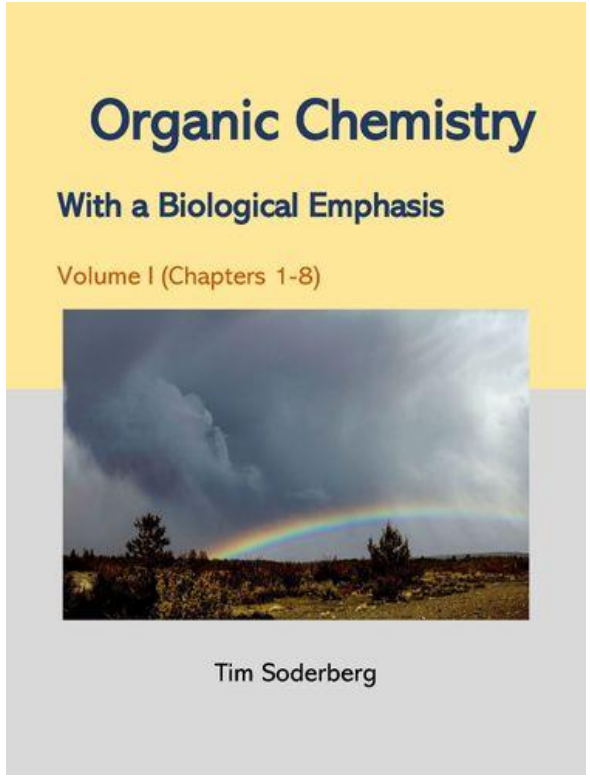

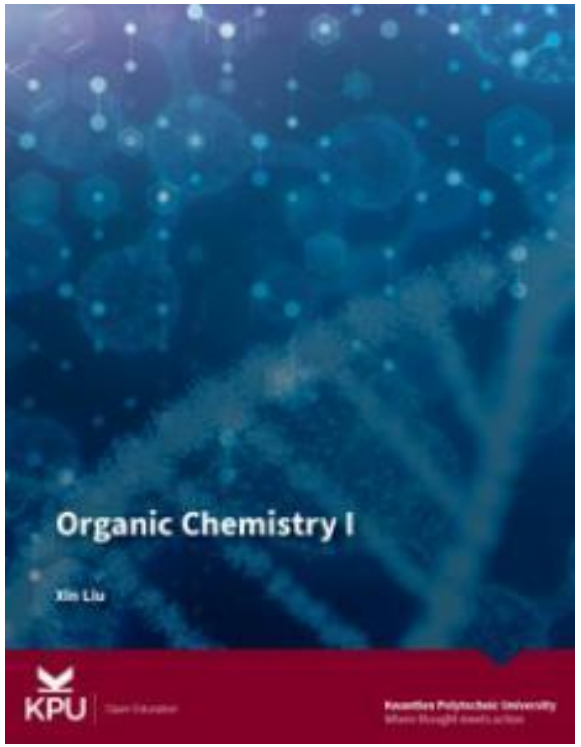

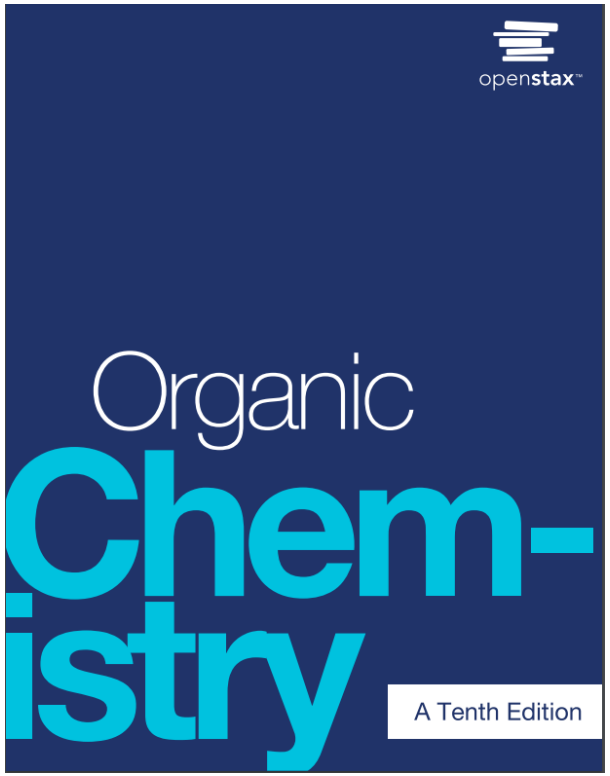

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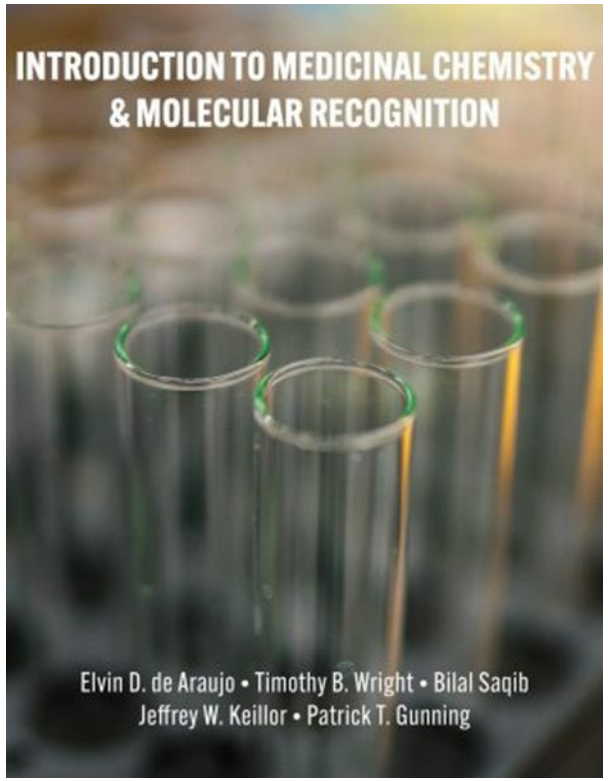
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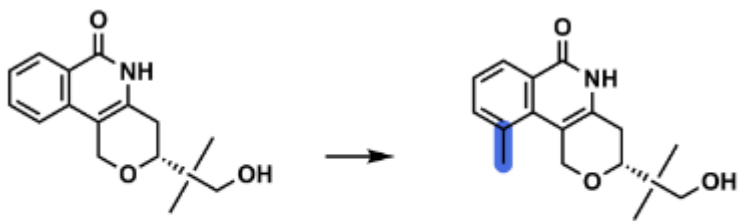
Jeffrey W. Keillor • Patrick T. Gunning

3.3 Binding is Determined by Drug : Target Interactions

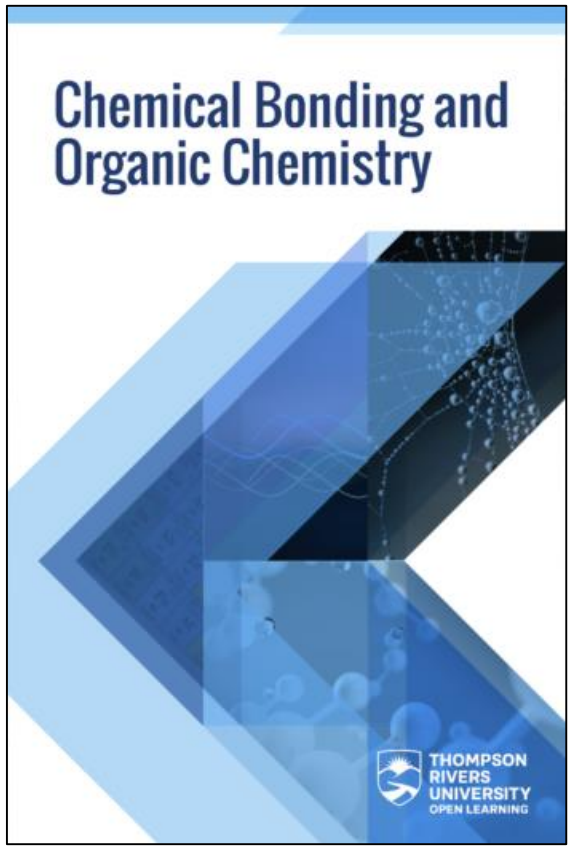
Ultimately, the capacity of a drug to engage with a target is determined by the thermodynamic binding energy. In a reversible reaction, the target enzyme (E) will bind an inhibitor (I) to give the complex E●I, where the equilibrium is governed by the constant K_d (or K_i in the presence of substrate). One of the main goals of medicinal chemistry and a significant portion of hit-to-lead investigations is to design molecules that will improve (decrease) the K_d. Understanding how to improve K_d, requires an understanding of the types of interactions that can occur between a target and its inhibitors. There are generally three types of binding interactions, i) electrostatic, ii) hydrogen bonding, and iii) "hydrophobic" interactions.



1
IC_{50} = 341 nM

2
IC_{50} = 7 nM

https://openintrochemistry.pressbooks.tru.ca/

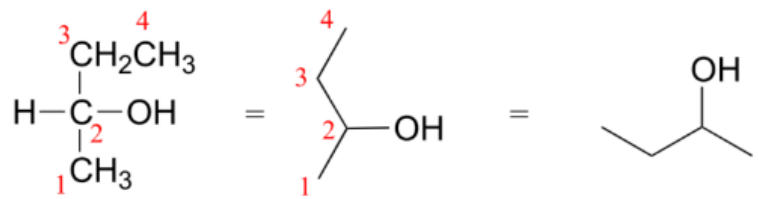


8.3 Stereochemistry of Organic Compounds and Pharmaceuticals

Chirality and Stereoisomers

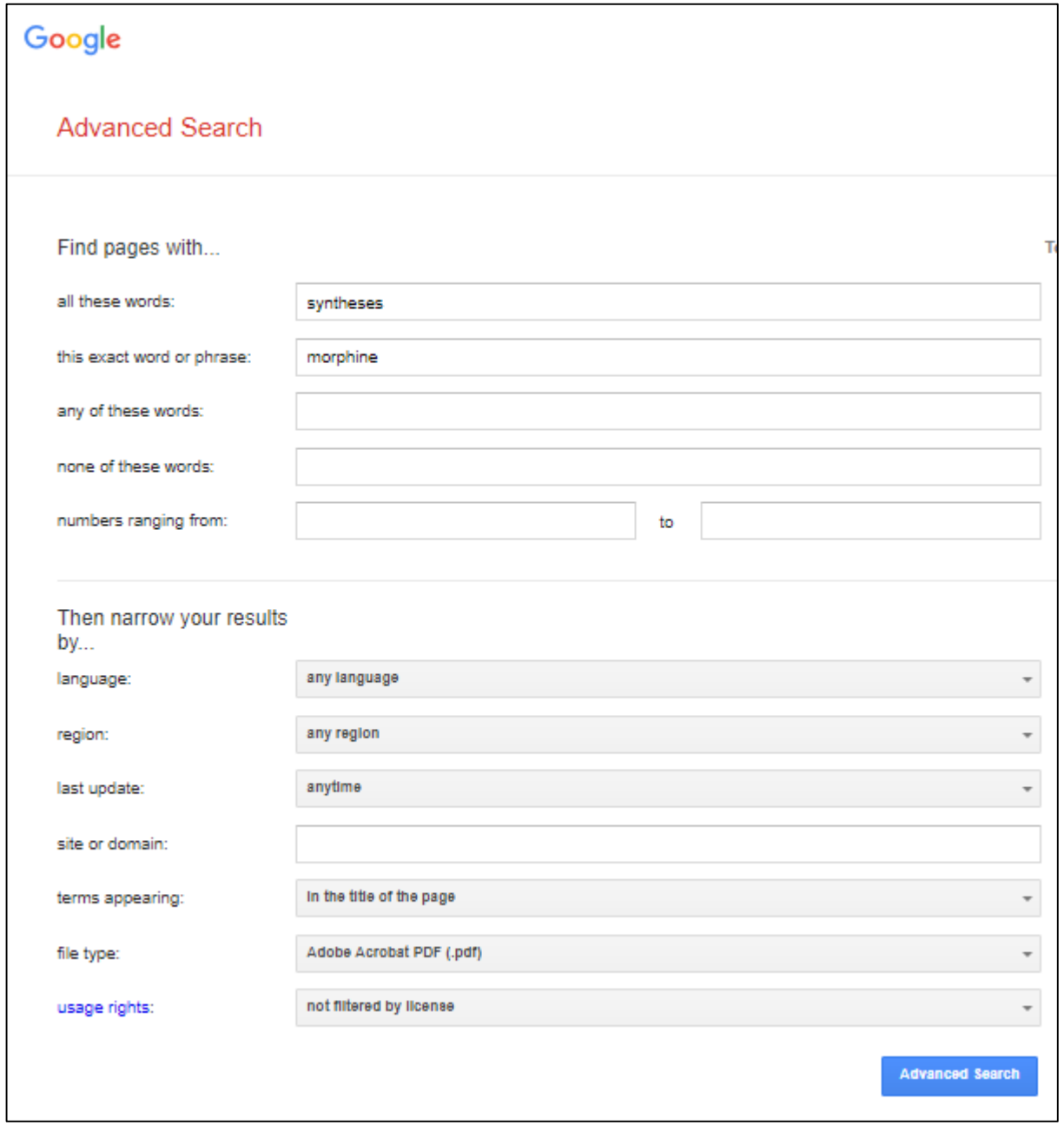
We turn now to the concept of chirality that formed the basis of the story about Louis Pasteur in the beginning of this chapter. Recall that the term **chiral**, from the Greek work for "hand," refers to anything which cannot be superimposed on its own mirror image. Your hands, of course, are chiral—you cannot superimpose your left hand on your right, and you cannot fit your left hand into a right-handed glove (which is also a ch
do not have a **mirror pl**
your hand in such a way
side. *Chiral objects do n*

Consider 2-butanol, drawn in two dimensions below.



2-butanol
(drawn in 2D)

https://www.google.ca/advanced_search



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Syntheses of Morphine

by M Maier · 1995 · Cited by 9 — Since drugs have been known, they have been contested in society. This is quite understand- able in the light of the misery and the crime.

People also ask

What is a synthetic version of morphine?

Can morphine be Synthesised?

What is a synthetic drug like morphine?

What is an opioid drug that is synthesized from morphine called?

Feedback

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Syntheses in the Direction of Morphine. II.1 Some ...

by MD Soffer · 1950 · Cited by 37 — bined, and the early literature is in many cases incomplete and con- fused. (8) Cornforth, Cornforth and Robinson, J. Chem. Soc,, 689. (1942).

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544. Syntheses in the Morphine Series. Part V.* Model

by D Elad · 1953 · Cited by 3 — IN Part IV * the synthesis of a tetracyclic compound (Ia) was described. This substance bears an oxygen function at C(5) which can be used as a key to…

Is there an open, AI-driven, small molecule, expert, chemical predictive retrosynthesis database?

Alternate Databases

Open Reaction Database
https://docs.open-reaction-database.org/

PubChem
https://pubchem.ncbi.nlm.nih.gov/

KEGG Reaction Database
https://www.genome.jp/kegg/reaction/

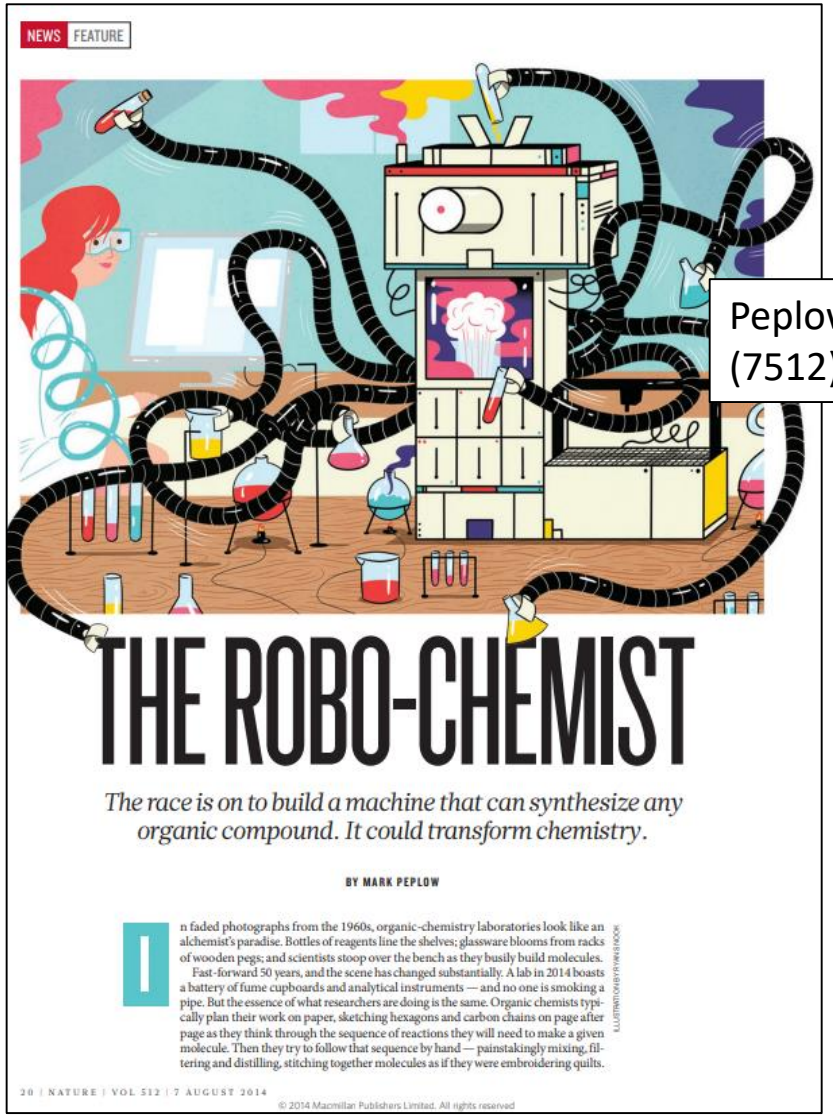
Organic Chemistry portal
https://www.organic-chemistry.org/

ChemSpider
https://www.chemspider.com/

Chemicalize
https://chemicalize.com/

ChEMBL
https://www.ebi.ac.uk/chembl/

Predictive Restrosynthesis Databases
https://www-library.ch.cam.ac.uk/list-useful-databases



NEWS FEATURE

THE ROBO-CHEMIST

The race is on to build a machine that can synthesize any organic compound. It could transform chemistry.

BY MARK PEPLOW

On faded photographs from the 1960s, organic-chemistry laboratories look like an alchemist's paradise. Bottles of reagents line the shelves; glassware blooms from racks of wooden pegs; and scientists stoop over the bench as they busily build molecules.

Fast-forward 50 years, and the scene has changed substantially. A lab in 2014 boasts a battery of fume cupboards and analytical instruments — and no one is smoking a pipe. But the essence of what researchers are doing is the same. Organic chemists typically plan their work on paper, sketching hexagons and carbon chains on page after page as they think through the sequence of reactions they will need to make a given molecule. Then they try to follow that sequence by hand — painstakingly mixing, filtering and distilling, stitching together molecules as if they were embroidering quilts.

ILLUSTRATION BY RYAN HUNTER

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Peplow, M. Organic Synthesis: The Robo-Chemist. Nature 2014, 512 (7512), 20–22. https://doi.org/10.1038/512020a.

https://www.nature.com/articles/512020a.pdf

Unfortunately, those failures are rarely recorded in the literature.

Money is also a significant hurdle.

“A synthesis machine would be transformational… [yet] I can see challenges”

Houben-Weyl Methods of Organic Chemistry

https://science-of-synthesis.thieme.com/app/search

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  - Science of Synthesis Backfile (more...)
  - Houben-Weyl (more...)

20.2.1.2.11 Synthesis of Carboxylic Acids from Carboxylic Acid Derivatives (Update 2024)

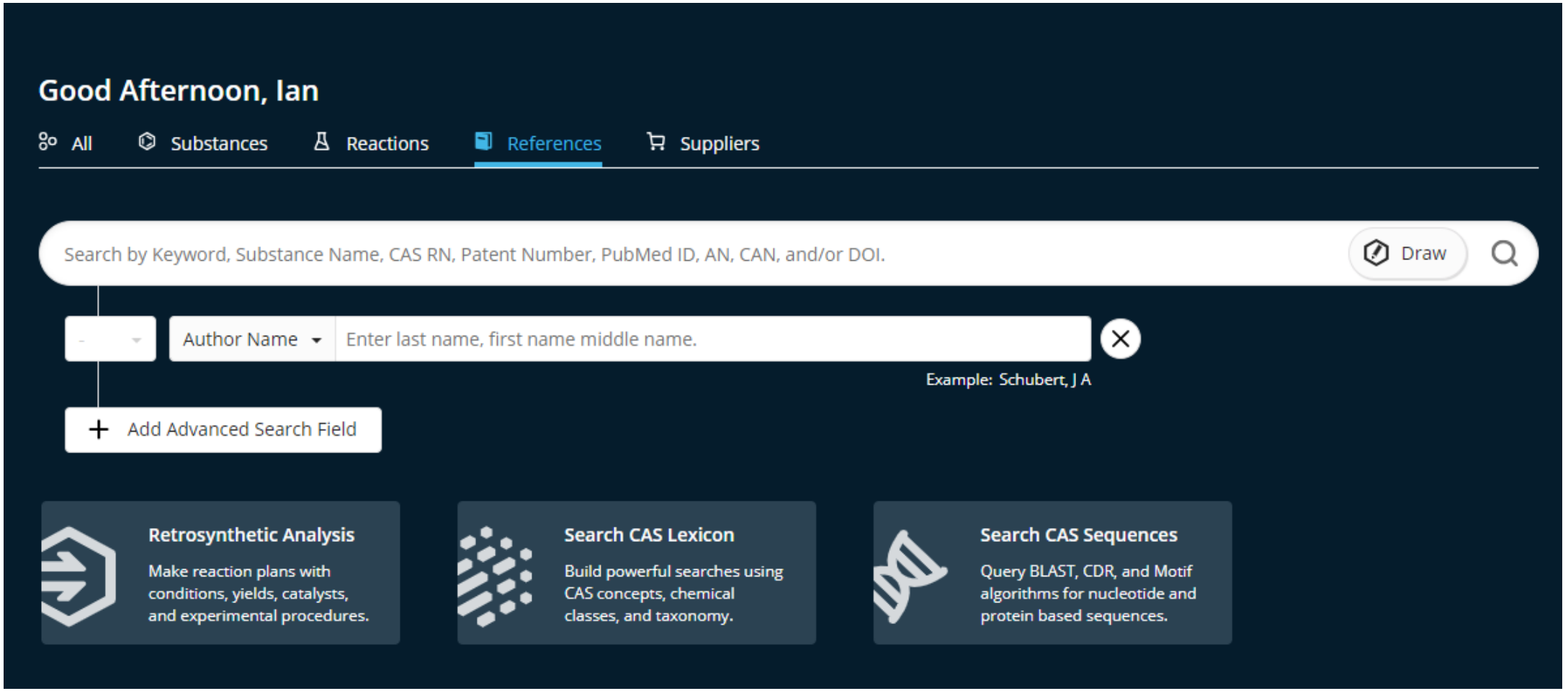
DOI: 10.1055/sos-SD-120-00336

Leclerc, E., *Science of Synthesis Knowledge Updates*, *early view*.

General Introduction

The synthesis of carboxylic acids from carboxylic acid derivatives has been described previously in *Science of Synthesis* (Section 20.2.1.2),[1] and an update to the original contribution on this topic was published in 2011 (Section 20.2.1.2.10).[2] The following sections describe either additional methods for known transformations, or the use of new precursors for the preparation of carboxylic acids that were not covered in the original chapter or the first update.

CAS SciFinder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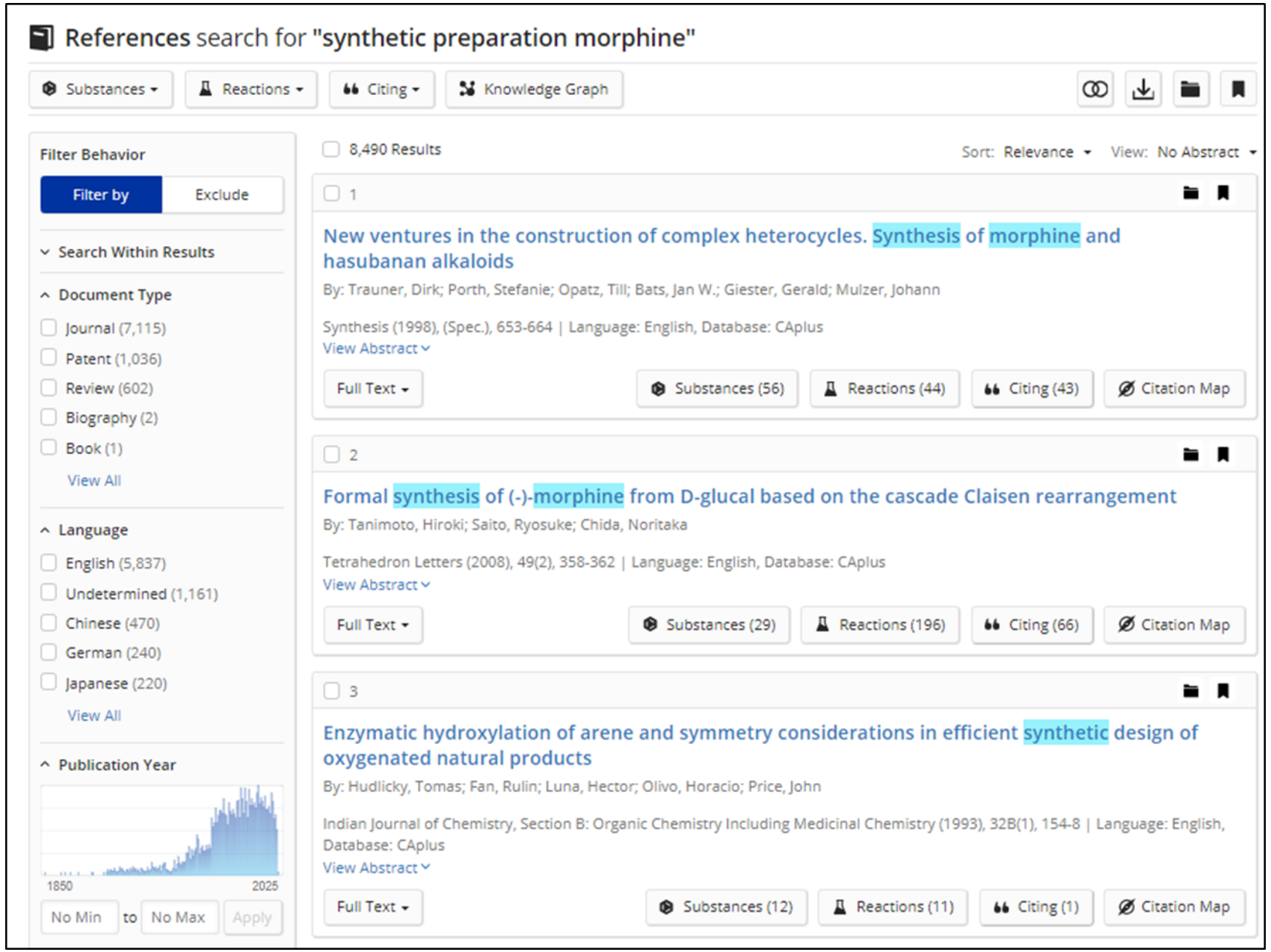
References search for "synthetic preparation morphine"
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Patent (1,036)
Review (602)
Biography (2)
Book (1)
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Undetermined (1,161)
Chinese (470)
German (240)
Japanese (220)
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1850
2025
No Min
to
No Max
Apply
8,490 Results
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View: No Abstract
1
New ventures in the construction of complex heterocycles. Synthesis of morphine and hasubanan alkaloids
By: Trauner, Dirk; Porth, Stefanie; Opatz, Till; Bats, Jan W.; Giester, Gerald; Mulzer, Johann
Synthesis (1998), (Spec.), 653-664 | Language: English, Database: CAplus
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Substances (56)
Reactions (44)
Citing (43)
Citation Map
2
Formal synthesis of (-)-morphine from D-glucal based on the cascade Claisen rearrangement
By: Tanimoto, Hiroki; Saito, Ryosuke; Chida, Noritaka
Tetrahedron Letters (2008), 49(2), 358-362 | Language: English, Database: CAplus
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Full Text
Substances (29)
Reactions (196)
Citing (66)
Citation Map
3
Enzymatic hydroxylation of arene and symmetry considerations in efficient synthetic design of oxygenated natural products
By: Hudlicky, Tomas; Fan, Rulin; Luna, Hector; Olivo, Horacio; Price, John
Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry (1993), 32B(1), 154-8 | Language: English, Database: CAplus
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Reactions (11)
Citing (1)
Citation Map

Morphine synthesis and biosynthesis-an update

1 | 0 | 94 | Citation Map

In this Reference

- CAS Concepts
- Substances
- Cited Documents

By: Novak, Bennett H.; Hudlicky, Tomas; Reed, Josephine W.; Mulzer, Johann; Trauner, Dirk

DOI: 10.2174/1385272003376292

This review with 106 references covers recent developments in the area of morphine synthesis and biosynthesis. Recent advancements in biosynthesis of morphine alkaloids are discussed. Total syntheses published since 1996 are reviewed and all published approaches to the morphine skeleton are discussed. This review includes an addnl. reference list for synthesis of medicinally important derivatives, improvements in alkaloid interconversion, and a list of all dissertations dealing with morphine synthesis.

Keywords: review morphine alkaloid derivative synthesis biosynthesis

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Reactions

44

31-614-CAS-25703138

Absolute stereochemistry shown, Rotation (-)

Absolute stereochemistry shown, Rotation (-)

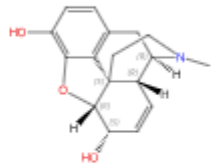
Steps: 1 **Yield:** 98%

View Experimental Protocols

1.1 Solvents: Dimethylformamide

Substances

57-27-2



Absolute stereochemistry shown, Rotation (-)

$C_{17}H_{19}NO_3$
Morphine

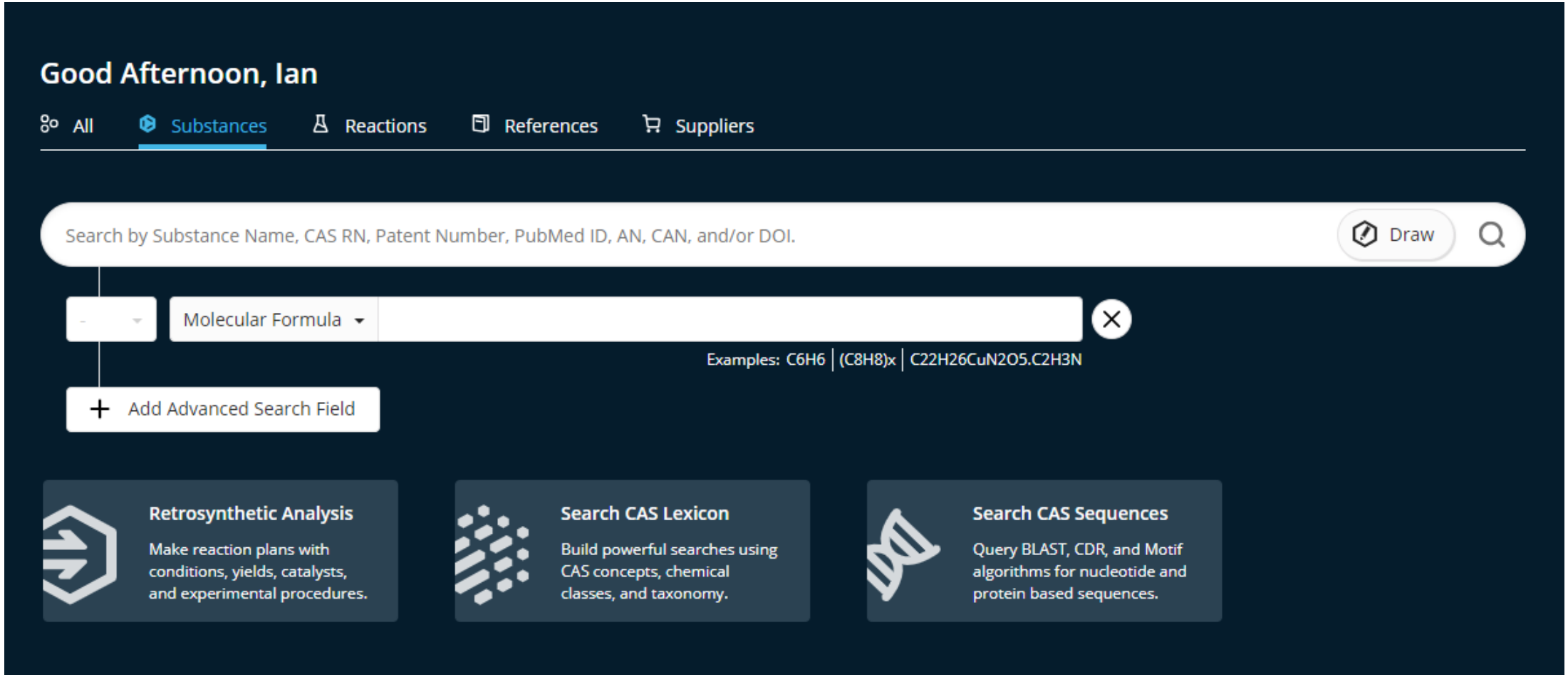
Role: Reactant, Synthetic Preparation, Reactant or Reagent, Preparation

Notes: derivatives

CAS Concepts

Alkaloids
Modifier: morphine-type
Role: Reactant; Synthetic Preparation

CAS SciFinder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Draw
Molecular Formula
Examples: C6H6 | (C8H8)x | C22H26CuN2O5.C2H3N
Add Advanced Search Field
Retrosynthetic Analysis
Make reaction plans with conditions, yields, catalysts, and experimental procedures.
Search CAS Lexicon
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CAS Draw

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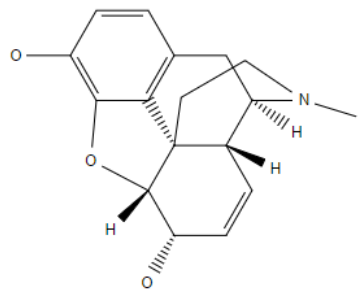
Draw ...oms or bonds.



Molecular Formula: $C_{17}H_{19}NO_3$ (285.34)

Taber, D. F.; Neubert, T. D.; Rheingold, A. L. Synthesis of (−)-Morphine. J. Am. Chem. Soc. **2002**, 124 (42), 12416–12417. https://doi.org/10.1021/ja027882h.

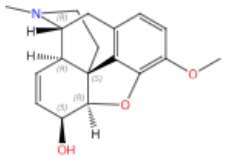
4

Synthesis of (-)-Morphine

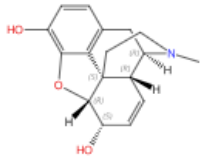
By: Taber, Douglass F.; Neubert, Timothy D.; Rheingold, Arnold L.

Journal of the American Chemical Society (2002), 124(42), 12416-12417 | Language: English, Database: CAplus and MEDLINE

Full Text



Absolute stereochemistry shown, Rotation (-)



Absolute stereochemistry shown, Rotation (-)

Suppliers (29)

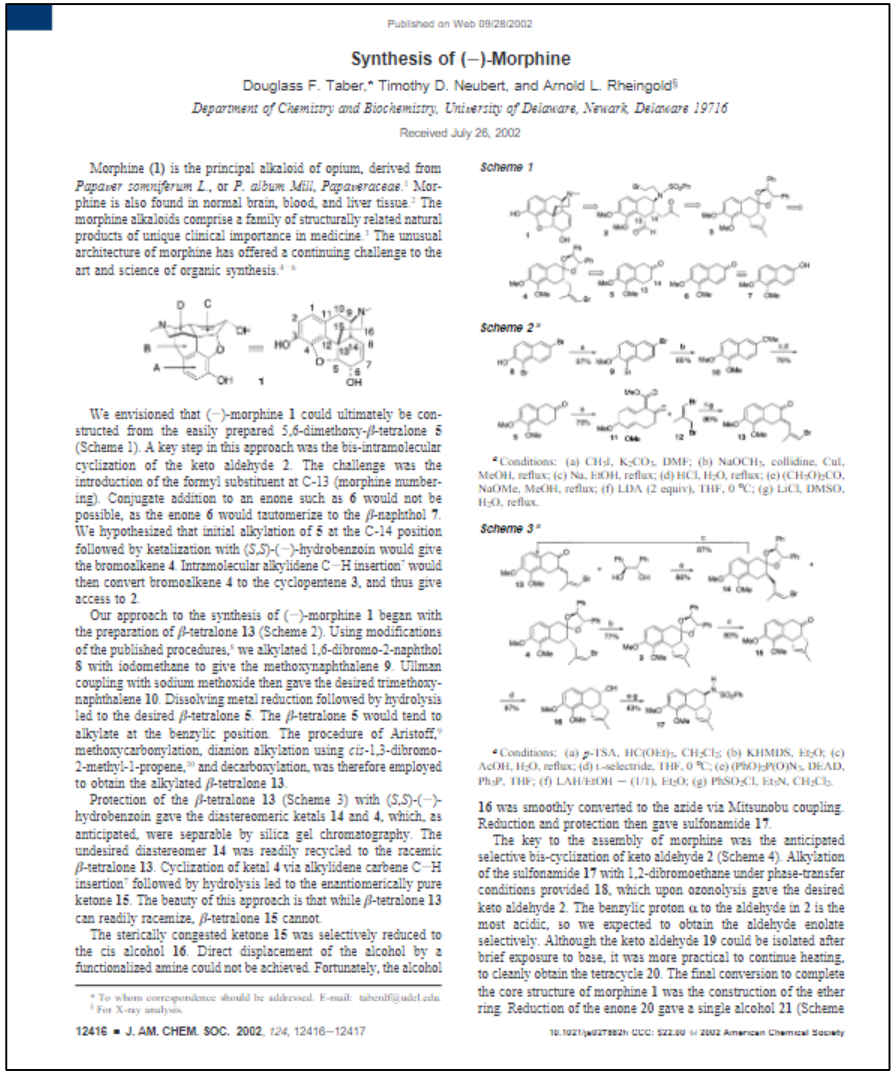
Suppliers (27)

31-049-CAS-13524928 Steps: 1 Yield: 86%

1.1
Reagents: Boron tribromide
Solvents: Dichloromethane

1.2
Reagents: Ammonium hydroxide
Solvents: Water

Experimental Protocols



Published on Web 09/28/2002

Synthesis of (−)-Morphine

Douglass F. Taber,* Timothy D. Neubert, and Arnold L. Rheingold§

Department of Chemistry and Biochemistry, University of Delaware, Newark, Delaware 19716

Received July 26, 2002

Morphine (**1**) is the principal alkaloid of opium, derived from *Papaver somniferum L.*, or *P. album Mill, Papaveraceae*.[1] Morphine is also found in normal brain, blood, and liver tissue.[2] The morphine alkaloids comprise a family of structurally related natural products of unique clinical importance in medicine.[3] The unusual architecture of morphine has offered a continuing challenge to the art and science of organic synthesis.[4,5]

We envisioned that (−)-morphine **1** could ultimately be constructed from the easily prepared 5,6-dimethoxy-β-tetralone **5** (Scheme 1). A key step in this approach was the bis-intramolecular cyclization of the keto aldehyde **2**. The challenge was the introduction of the formyl substituent at C-13 (morphine numbering). Conjugate addition to an enone such as **6** would not be possible, as the enone **6** would tautomerize to the β-naphthol **7**. We hypothesized that initial alkylation of **5** at the C-14 position followed by ketalization with (*S,S*)-(−)-hydrobenzoin would give the bromoalkene **4**. Intramolecular alkylidene C−H insertion[6] would then convert bromoalkene **4** to the cyclopentene **3**, and thus give access to **2**.

Our approach to the synthesis of (−)-morphine **1** began with the preparation of β-tetralone **13** (Scheme 2). Using modifications of the published procedures,[8] we alkylated 1,6-dibromo-2-naphthol **8** with iodomethane to give the methoxynaphthalene **9**. Ullman coupling with sodium methoxide then gave the desired trimethoxynaphthalene **10**. Dissolving metal reduction followed by hydrolysis led to the desired β-tetralone **5**. The β-tetralone **5** would tend to alkylate at the benzylic position. The procedure of Aristoff,[9] methoxycarbonylation, dianion alkylation using *cis*-1,3-dibromo-2-methyl-1-propene,[10] and decarboxylation, was therefore employed to obtain the alkylated β-tetralone **13**.

Protection of the β-tetralone **13** (Scheme 3) with (*S,S*)-(−)-hydrobenzoin gave the diastereomeric ketals **14** and **4**, which, as anticipated, were separable by silica gel chromatography. The undesired diastereomer **14** was readily recycled to the racemic β-tetralone **13**. Cyclization of ketal **4** via alkylidene carbene C−H insertion[7] followed by hydrolysis led to the enantiomerically pure ketone **15**. The beauty of this approach is that while β-tetralone **13** can readily racemize, β-tetralone **15** cannot.

The sterically congested ketone **15** was selectively reduced to the cis alcohol **16**. Direct displacement of the alcohol by a functionalized amine could not be achieved. Fortunately, the alcohol

* To whom correspondence should be addressed. E-mail: taberdf@udel.edu.
§ For X-ray analysis.

10.1021/ja027882h CCC: $22.00 © 2002 American Chemical Society

Scheme 1

Scheme 2[a]

[a] Conditions: (a) CH_2I_2, K_2CO_3, DMF; (b) $NaOCH_3$, collidine, CuI, MeOH, reflux; (c) Na, EtOH, reflux; (d) HCl, H_2O, reflux; (e) $(CH_3O)_2CO$, NaOMe, MeOH, reflux; (f) LDA (2 equiv), THF, 0 °C; (g) LiCl, DMSO, H_2O, reflux.

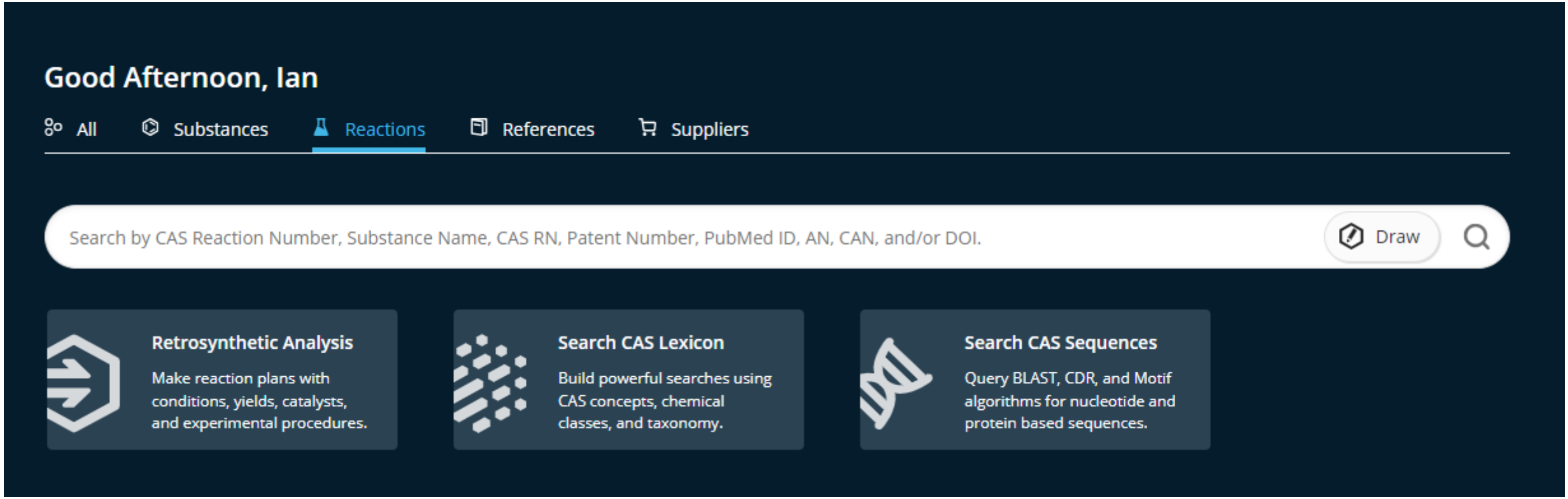
Scheme 3[a]

[a] Conditions: (a) p-TSA, $HC(OEt)_3$, CH_2Cl_2; (b) KHMDS, Et_2O; (c) AcOH, H_2O, reflux; (d) L-selectride, THF, 0 °C; (e) $(PhO)_2P(O)N_3$, DEAD, Ph_3P, THF; (f) LAH/EtOH − (1/1), Et_2O; (g) $PhSO_2Cl$, Et_3N, CH_2Cl_2.

16 was smoothly converted to the azide via Mitsunobu coupling. Reduction and protection then gave sulfonamide **17**.

The key to the assembly of morphine was the anticipated selective bis-cyclization of keto aldehyde **2** (Scheme 4). Alkylation of the sulfonamide **17** with 1,2-dibromoethane under phase-transfer conditions provided **18**, which upon ozonolysis gave the desired keto aldehyde **2**. The benzylic proton α to the aldehyde in **2** is the most acidic, so we expected to obtain the aldehyde enolate selectively. Although the keto aldehyde **19** could be isolated after brief exposure to base, it was more practical to continue heating, to cleanly obtain the tetracycle **20**. The final conversion to complete the core structure of morphine **1** was the construction of the ether ring. Reduction of the enone **20** gave a single alcohol **21** (Scheme

CAS SciFinder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Reactions for 57-27-2

References ▾ | Save ▾

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Substance Role

- [x] Product (122)

Yield

- [ ] No Yield Available (122)

Number of Steps

- [ ] 1 (40)
- [x] 2 (25)
- [x] 3 (27)
- [x] 4 (33)
- [x] 5 (37)
- [ ] 6-10 (331)
- [ ] 11-15 (563)
- [ ] 16-20 (129)
- [ ] >20 (6)

View Fewer

Filtering: Substance Role: **Product** ✕ | Number of Steps: 4 Selected ▾ ✕ | Clear All Filters

☐ 122 Results | Group: By Scheme ▾ | Sort: Yield ▾ | View: Expanded ▾

Scheme 1 (1 Reaction) Steps: 2 •••

Absolute stereochemistry shown → Absolute stereochemistry shown, Rotation (-)

Suppliers (27)

☐ View Reaction Detail Steps: 2

1.1 Reagents: Lithium aluminum hydride
Solvents: Tetrahydrofuran
2.1 Reagents: Pyridinium chloride

Synthesis of metabolites of 3-O-tert-butylmorphine

By: Mohacsi, E.

Journal of Heterocyclic Chemistry (1987), 24(2), 471-2

Full Text ▾

Collapse Scheme ^

Scheme 2 (2 Reactions) Steps: 2 •••

CAS Reaction Number: 31-614-CAS-25703138

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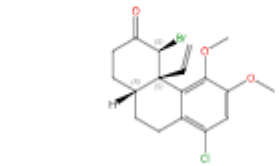
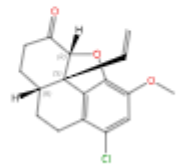


Absolute stereochemistry shown, Rotation (-)



Absolute stereochemistry shown, Rotation (-)

98%

Experimental Protocols

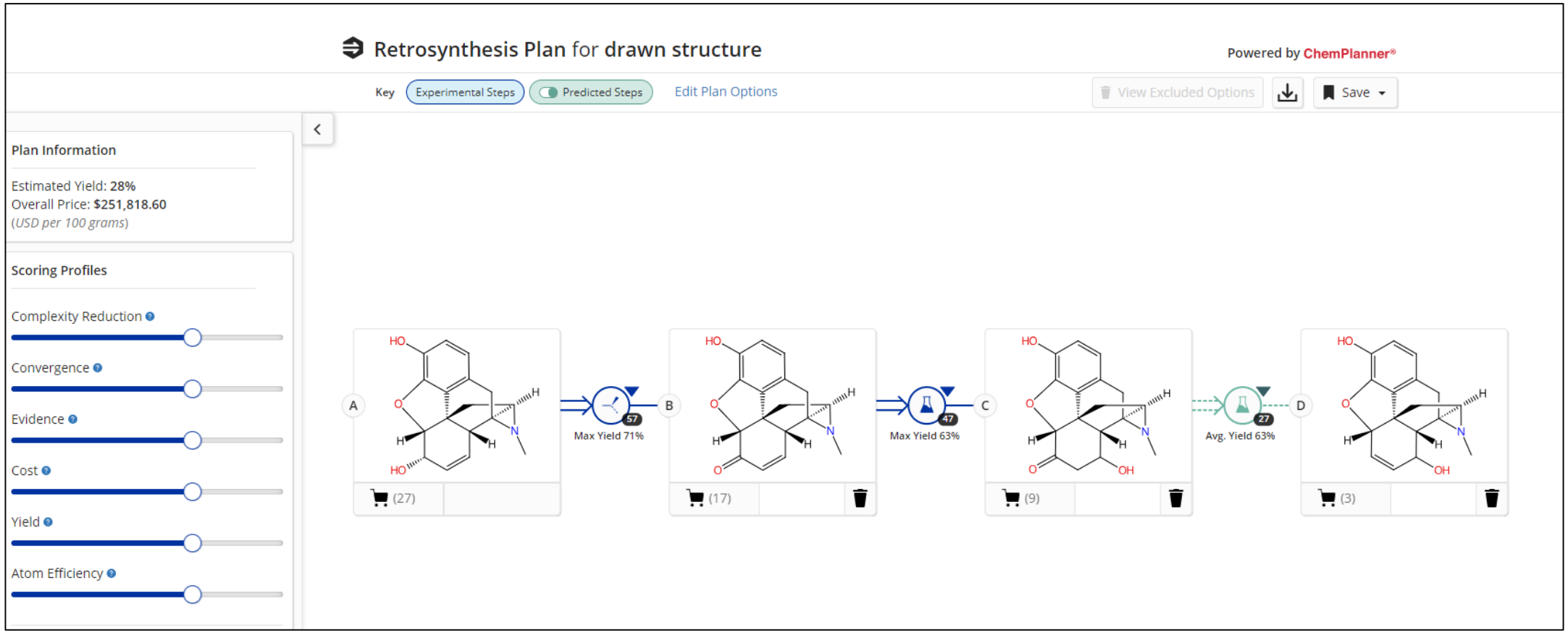
Synthetic Methods | Experimental Procedure

Products	Phenanthro[4,5-*bcd*]furan-3(2*H*)-one, 7-chloro-9b-ethenyl-1,3a,8,9,9a,9b-hexahydro-5-methoxy-, (3a*R*,9a*R*,9b*S*)-, Yield: 98%
Reactants	(4*S*,4a*S*,10a*R*)-4-Bromo-8-chloro-4a-ethenyl-1,4,4a,9,10,10a-hexahydro-5,6-dimethoxy-3(2*H*)-phenanthrenone
Solvents	Dimethylformamide
Procedure	1. Heat a solution of bromoketone derivative (3.99 g) in anhydrous DMF (50 mL) to 140°C for 20 minutes. 2. Cool the solution to room temperature. 3. Remove the solvent in vacuo. 4. Purify the crystalline crude product by chromatography on silica gel, eluting with hexane/EtOAc (1/1).
Scale	gram

Characterization Data

^ Phenanthro[4,5-*bcd*]furan-3(2*H*)-one, 7-chloro-9b-ethenyl-1,3a,8,9,9a,9b-hexahydro-5-methoxy-, (3a*R*,9a*R*,9b*S*)-

Proton NMR Spectrum	250 MHz, $CDCl_3$: δ (dq, 1 H, *J*= 4.9, 12.6 Hz), 1.70-2.20 (m, 3 H), 2.23-2.45 (m, 4 H), 2.61 (ddd, 1 H, *J*= 0.9, 7.3, 18.0 Hz), 3.82 (s, 3 H), 4.52 (dd, 1 H, *J*= 0.7, 17.0 Hz), 4.71 (s, 1 H), 5.14 (dd, 1 H, *J*= 0.7, 10.2 Hz), 5.97 (dd, 1 H, *J*= 10.2, 17.0 Hz), 6.71 (s, 1 H).
Carbon-13 NMR	63 MHz, $CDCl_3$: δ 22.9, 26.5, 36.1, 39.4, 55.2, 56.6, 91.7, 114.7, 117.1, 123.9, 125.0, 126.8, 141.8, 142.9, 145.6, 206.5.
IR Absorption Spectrum	Si: ν = 2938, 1729, 1631, 1491, 1436, 1186, 1147, 1096, 1037, 973, 873 cm^{-1}.
Optical Rotatory Power	$[\alpha]_D$ -35.7, (*c* = 2, $CHCl_3$).
Elemental Analysis	$C_{17}H_{17}ClO_3$ (304.8): calcd C, 67.00; H, 5.62; found C, 66.92; H, 5.55.
HRMS	*m/z*calcd for $C_{17}H_{17}ClO_3$ 304.08662, found: 304.08662 ± 0.0015.
Mass Spectrum	EI, 70 eV, 30°C: *m/z*(%) = 306 (57), 304 ($M^{+\cdot}$, 99), 244 (87), 174 (76), 149 (28), 131 (41), 95 (33), 69 (100).
Melting Point	147-148 °C (racemic), 178-179°C (enantio-merically pure).
R_f	0.25 (hexane/EtOAc, 3:1).
State	colorless crystals.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A
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Max Yield 63%
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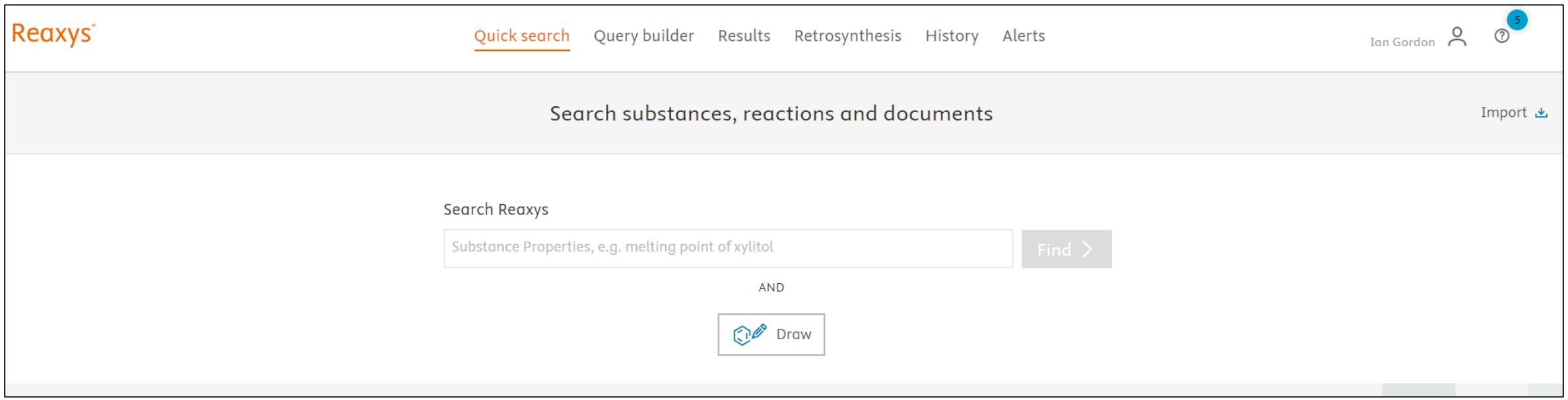
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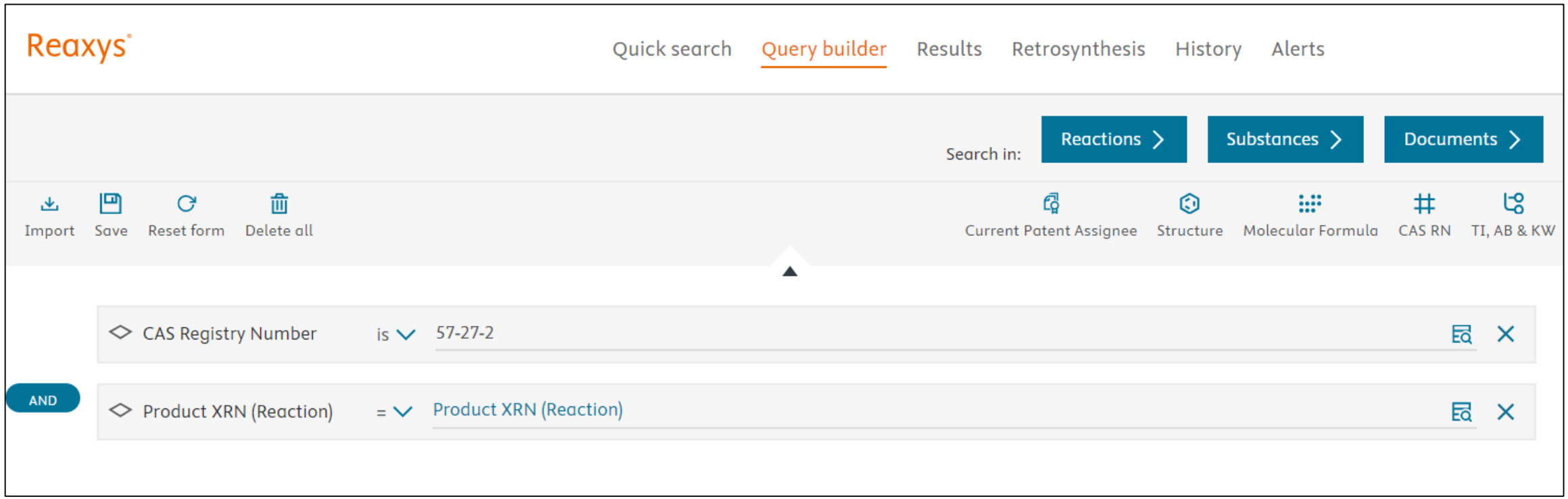
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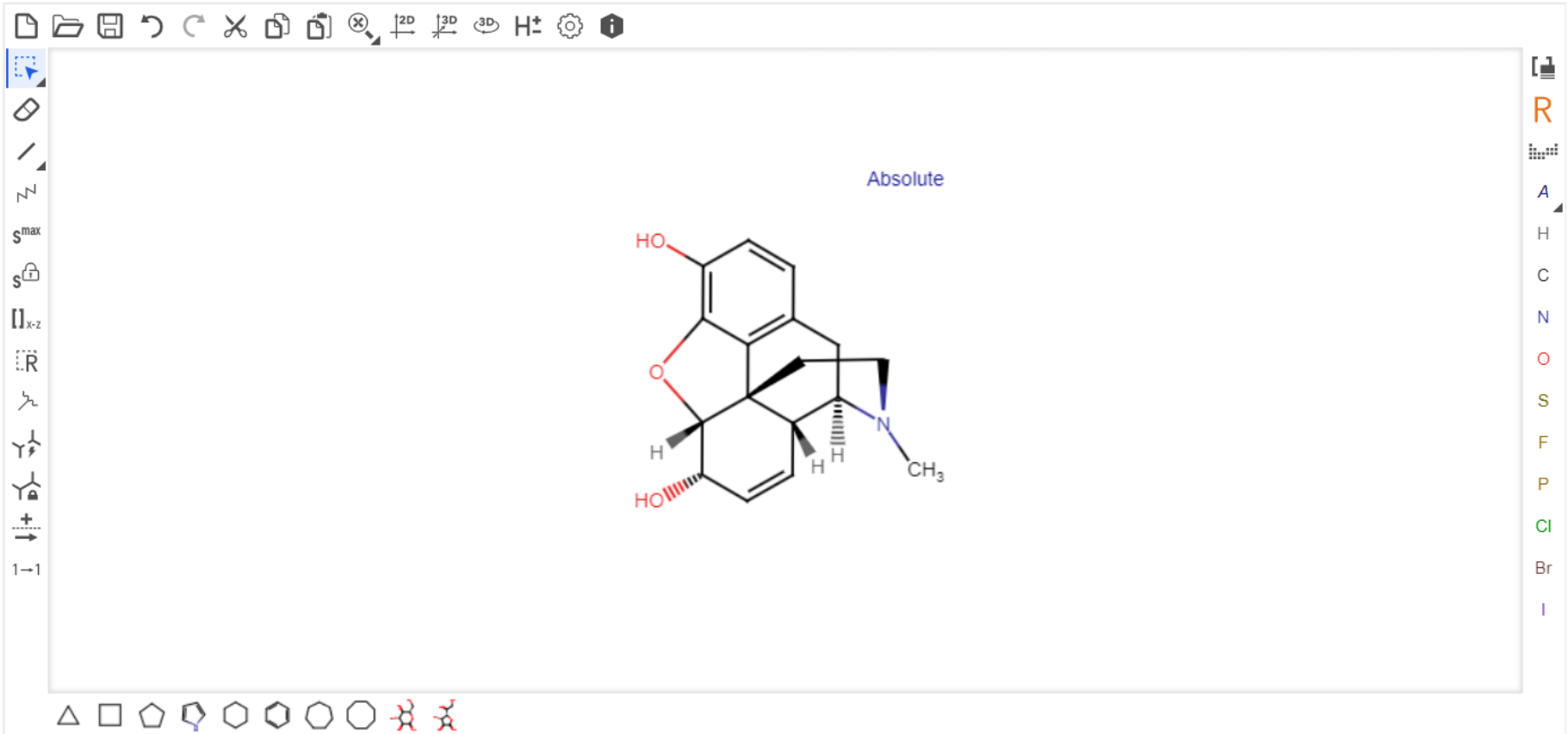

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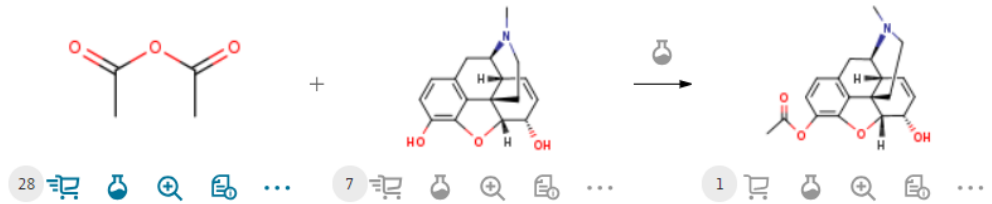

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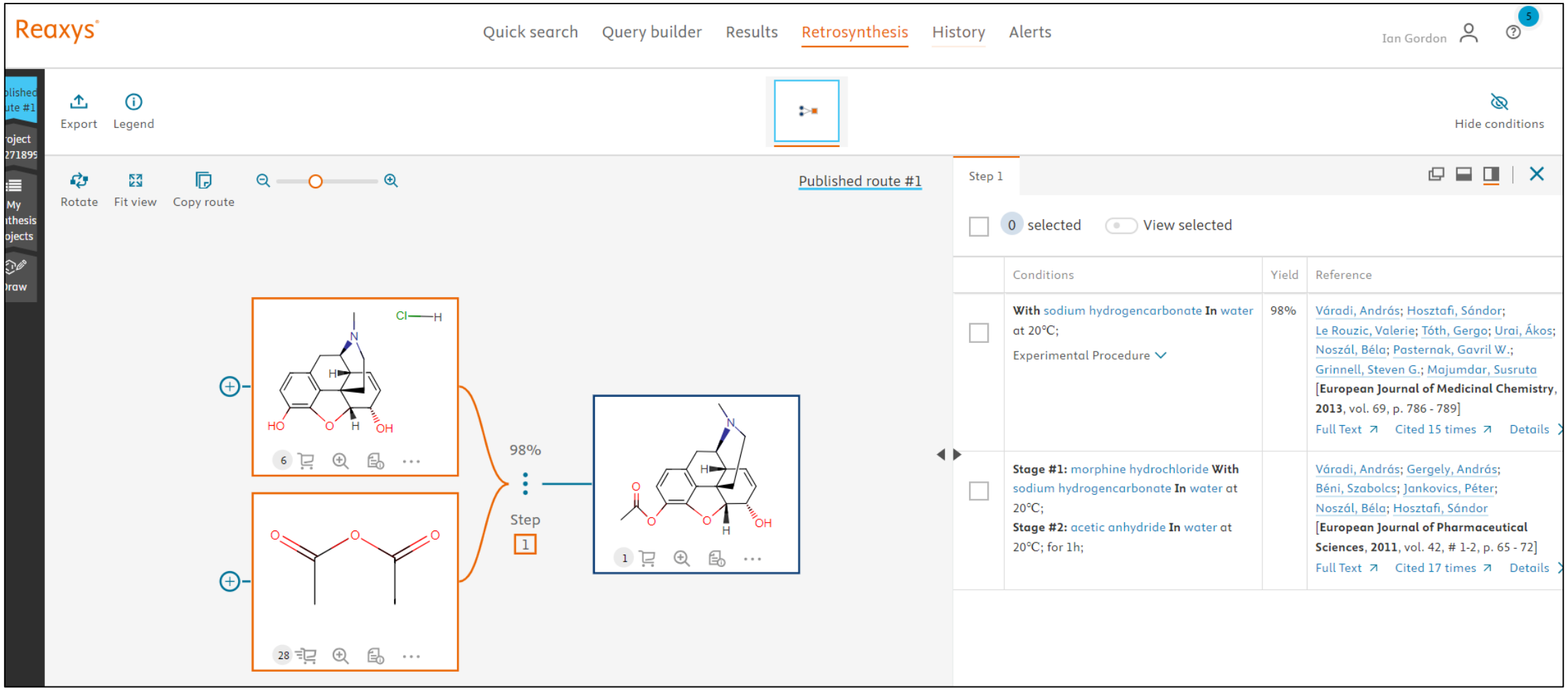
9 Conditions Find Similar > Reaction ID: 832970

Conditions	Yield	Reference
With sodium hydrogencarbonate **In** water at 20°C; for 0.25h; Experimental Procedure	99%	Ahonen, Tiina J.; Rinne, Maiju; Grutschreiber, Peter; Mätlik, Kert; Airavaara, Mikko; Schaarschmidt, Dieter; Lang, Heinrich; (...) Yli-Kauhaluoma, Jari; Moreira, Vânia M. [**European Journal of Medicinal Chemistry**, **2018**, vol. 151, p. 495 - 507] Full Text Cited 3 times Details Abstract
With phosphate buffer pH=7; Acetylation;	90%	Berrang, Bertold D.; Wyrick, Christopher D.; Carroll, F. Ivy; Seltzman, Herbert H. [**Journal of labelled compounds and radiopharmaceuticals**, **1999**, vol. 42, # 9, p. 851 - 857] Full Text Cited 5 times Details Abstract

1

1 Conditions Find Similar Reaction ID: 20800136

Conditions	Yield	Reference
Multi-step reaction with 11 steps 1: 77 percent / H_2 / 5 percent Pd/C / ethyl acetate / 735.5 Torr 2: 1.) pyridine / 1.) 0 deg C, 1 h; 2.) water, 30 min 3: 39 g / imidazole / dimethylformamide; CH_2Cl_2 / 1.) RT, 2 h; 2.) 45 deg C, 20 h; 3.) 80 deg C, 3 h 4: 82.6 percent / 85 percent KOH / methanol; benzene / 2.5 h / Ambient temperature 5: 96 percent / pyridinium chlorochromate / CH_2Cl_2 / 1 h 6: 63 percent / tetrahydrofuran; CH_2Cl_2 / 1.5 h / -78 °C 7: 99 percent / 4-N,N-dimethylaminopyridine / pyridine / 1.5 h 8: 1.) ozone; 2.) dimethylsulfide / 1.) MeOH, -78 deg C, 10 min; 2.) -10 deg C, 1 h, 0 deg C 1 h, RT, 1 h 9: 95 percent / Et_3N / CH_2Cl_2 / 40 h / Ambient temperature 10: 81 percent / 42 h / 50 °C 11: 82 percent / 4.4M NaOMe / methanol; tetrahydrofuran / 28.5 h View Scheme		Wovkulich; Barcelos; Batcho; et al. [**Tetrahedron**, **1984**, vol. 40, # 12, p. 2283 - 2296] Full Text Cited 87 times Details Abstract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Step 1
0 selected
View selected
Conditions
Yield
Reference
With sodium hydrogencarbonate In water at 20°C;
Experimental Procedure
98%
Váradi, András; Hosztafi, Sándor; Le Rouzic, Valerie; Tóth, Gergo; Urai, Ákos; Noszál, Béla; Pasternak, Gavril W.; Grinnell, Steven G.; Majumdar, Susruta [European Journal of Medicinal Chemistry, 2013, vol. 69, p. 786 - 789]
Full Text
Cited 15 times
Details
Stage #1: morphine hydrochloride With sodium hydrogencarbonate In water at 20°C;
Stage #2: acetic anhydride In water at 20°C; for 1h;
Váradi, András; Gergely, András; Béni, Szabolcs; Jankovics, Péter; Noszál, Béla; Hosztafi, Sándor [European Journal of Pharmaceutical Sciences, 2011, vol. 42, # 1-2, p. 65 - 72]
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Step
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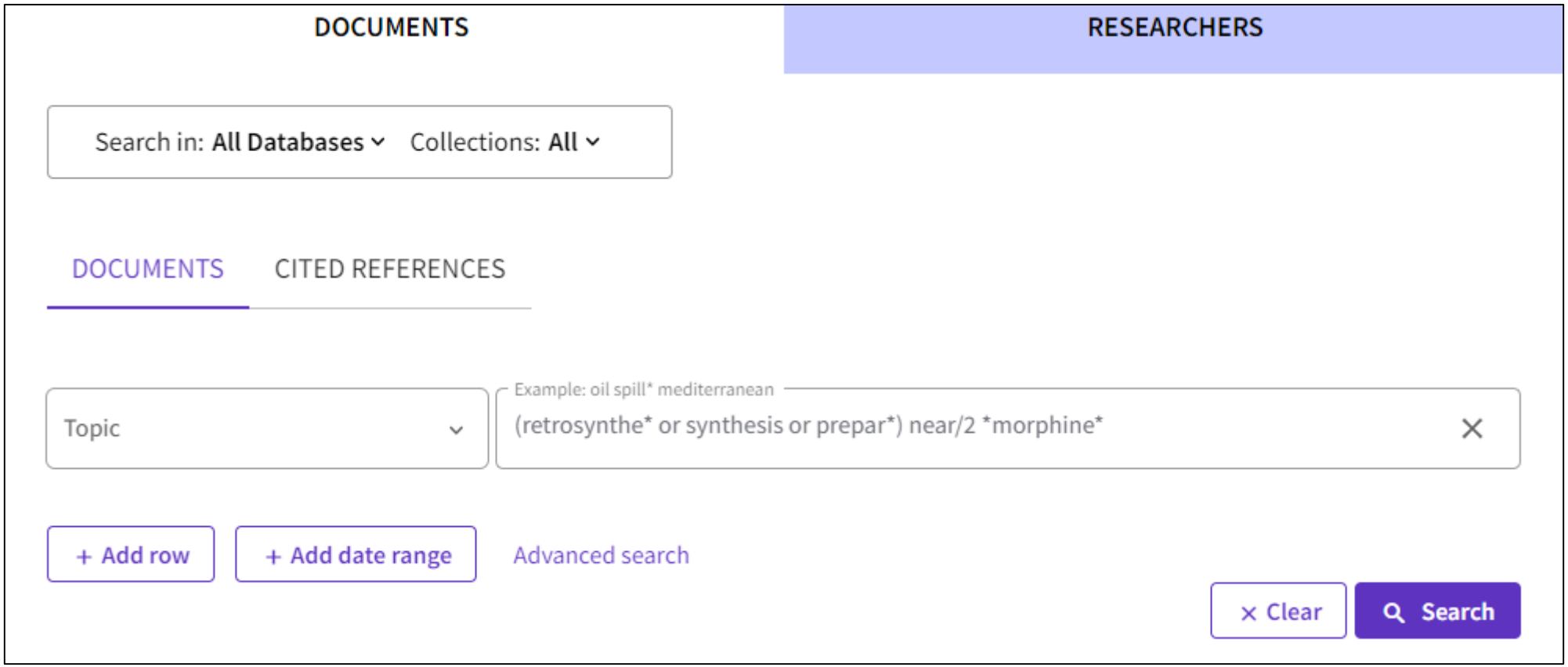

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PurposeOral Morphine Equivalent (OME) doses are increasingly being used as a metric to represent opioid use. Driven by a growing need from pharmacoepidemiological studies, the objective of this study was to develop a comprehensive OME conversion table that can be used by researchers to calculate OMEs in a consistent and systematic way ... Show more

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Dec 2004 | NATURE BIOTECHNOLOGY 22 (12) , pp.1559-1566

260 Citations

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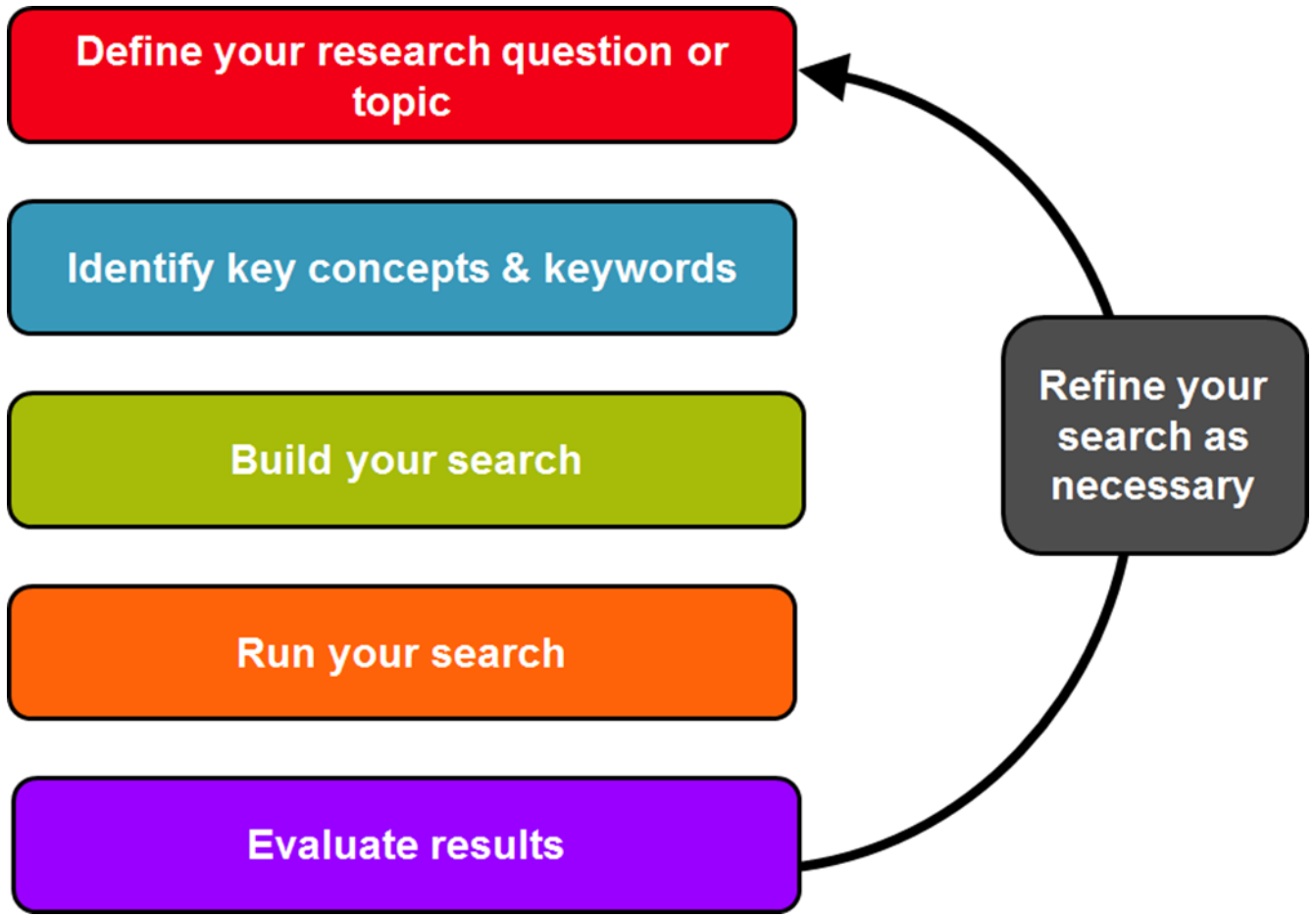
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- Citation management
- Where, how and when to get help

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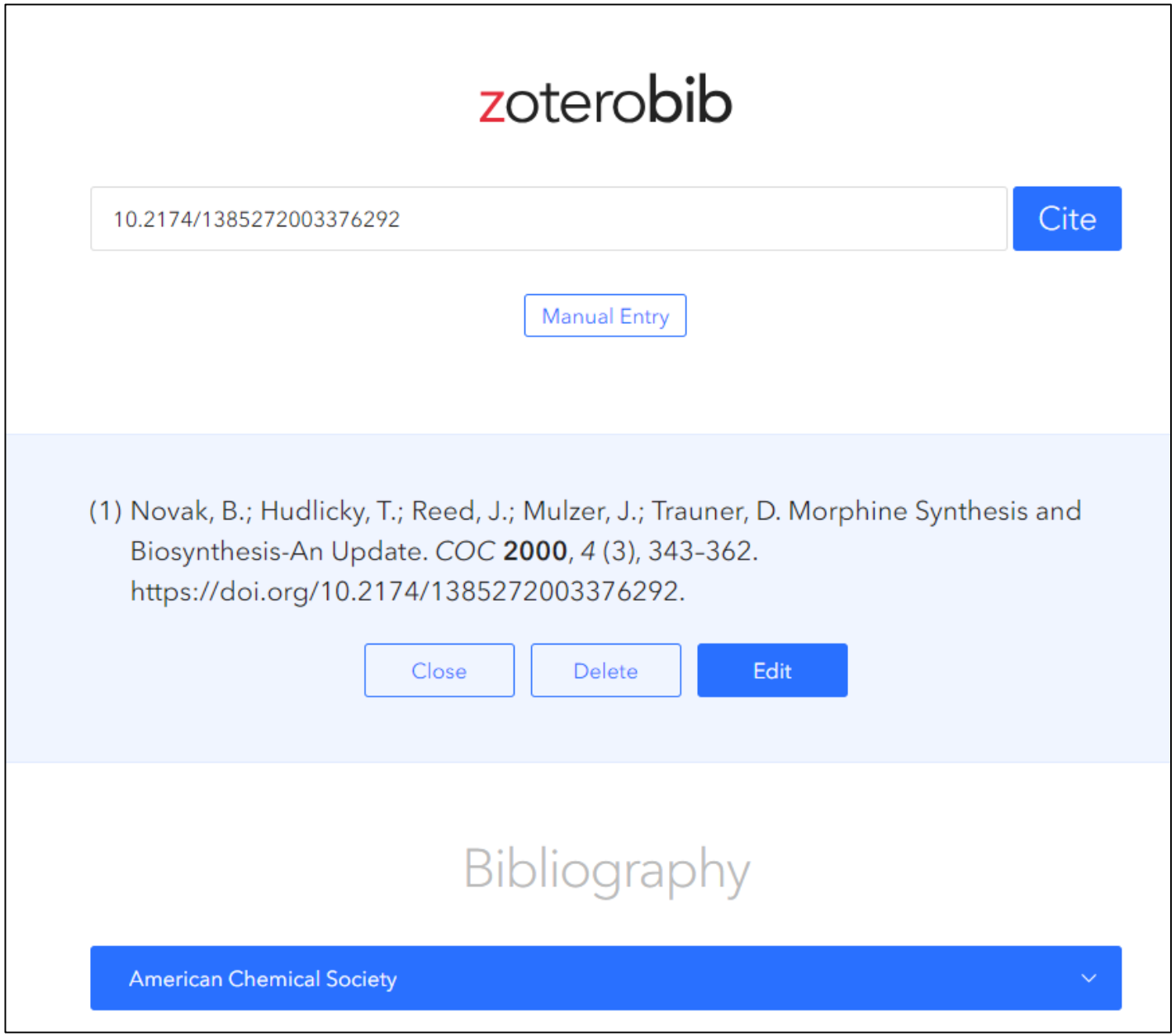

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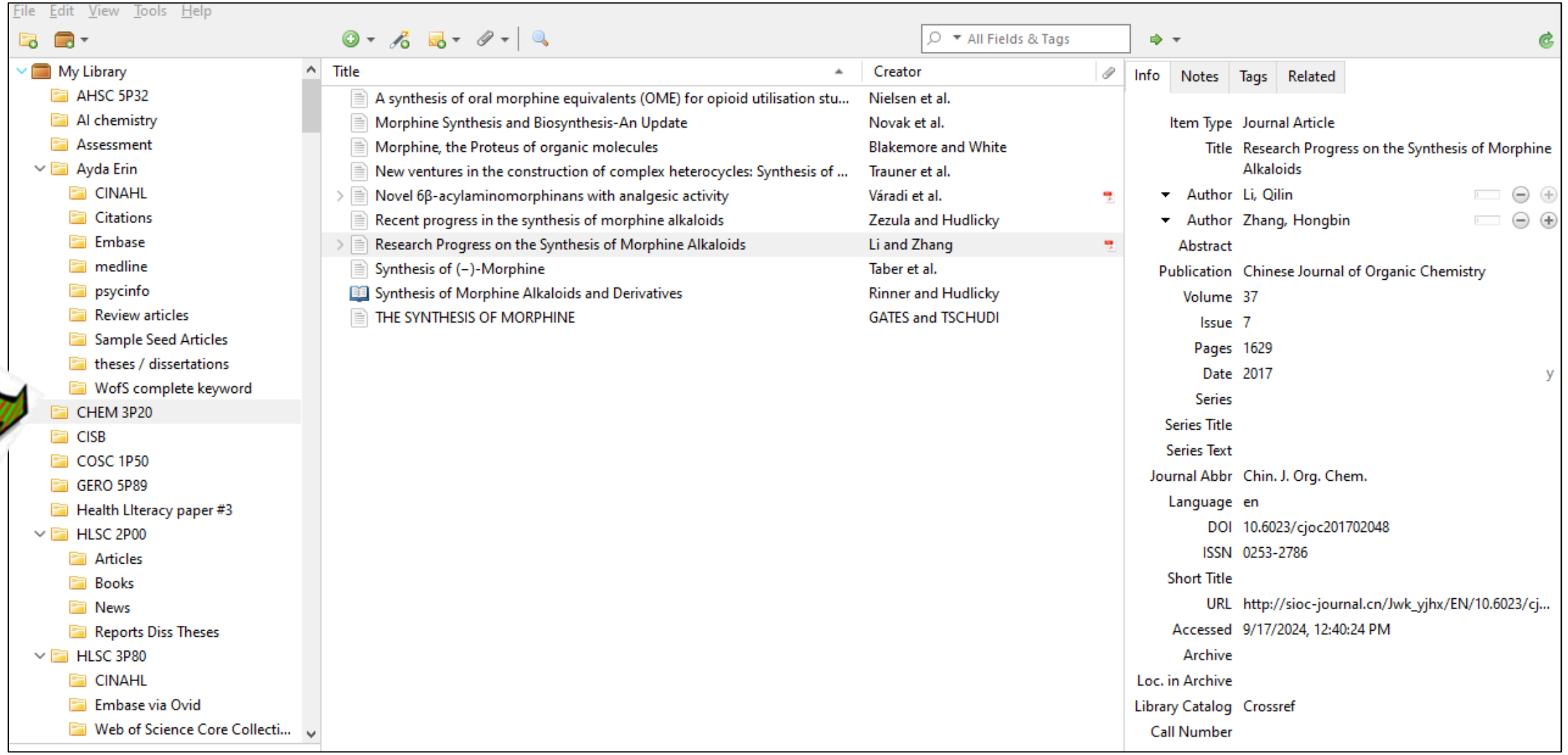

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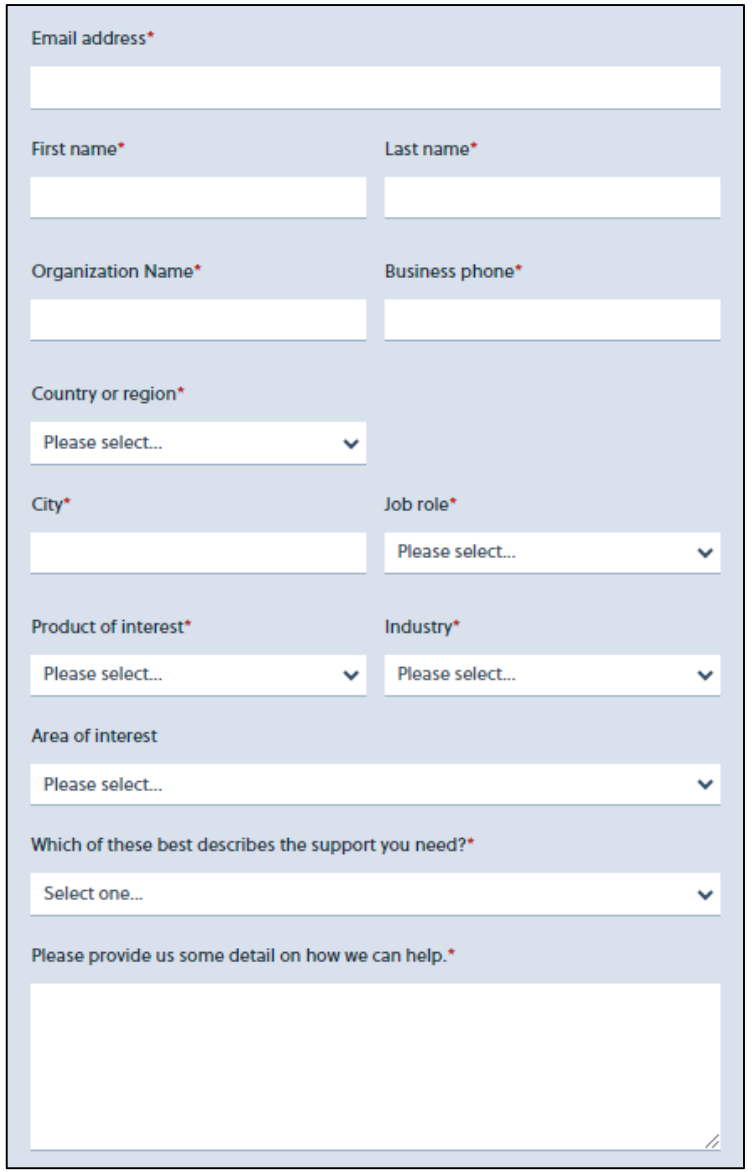

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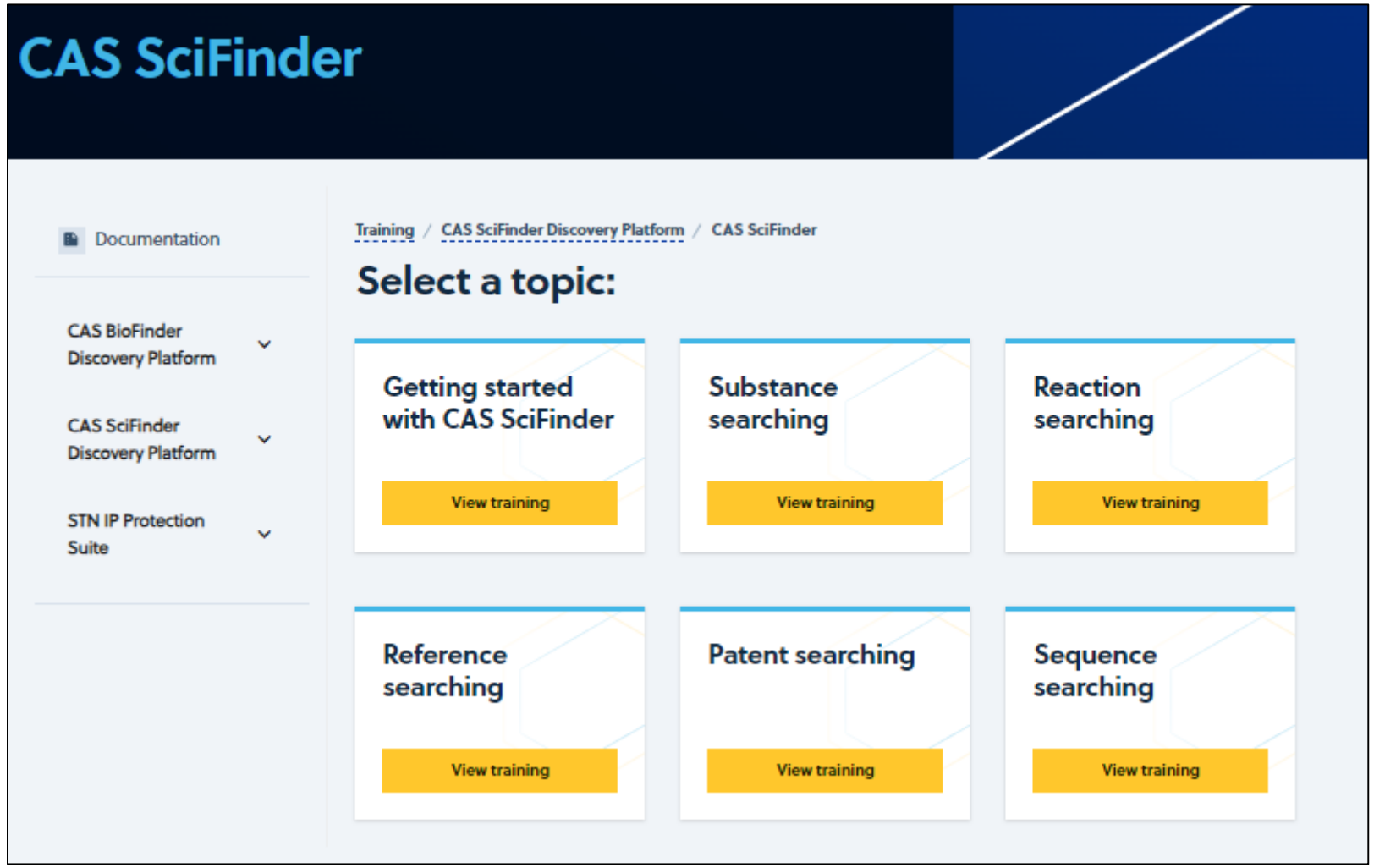

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